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WADC TECHNICAL REPORT 53-373  
SUPPLEMENT 1  
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## A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

MARY M. SOKAS, 1ST LT, USAF

MATERIALS LABORATORY

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DECEMBER 1954

Statement A  
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WRIGHT AIR DEVELOPMENT CENTER

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WADC TECHNICAL REPORT 53-373  
SUPPLEMENT 1

# **A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM**

*MARY M. SOKAS, 1ST LT, USAF*

*MATERIALS LABORATORY*

*DECEMBER 1954*

WRIGHT AIR DEVELOPMENT CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

## FOREWORD

This report was prepared by Mary M. Sokas, 1st Lt, USAF, Chief, Technical Data Section, Technical Services Branch, Materials Laboratory, Directorate of Research, Wright Air Development Center, Wright-Patterson Air Force Base.

The assistance of Mrs. Annie Rainey in the preparation of this report is gratefully acknowledged.

The Technical Reports referenced herein cover the period 1 July 1953-30 June 1954 and have been released to the Armed Services Technical Information Agency.

## ABSTRACT

One hundred and thirty (130) technical reports issued during the period 1 July 1953 - 30 June 1954 are abstracted. These reports cover the areas of metallurgy, textiles, petroleum products, structural materials, rubbers, plastics, packaging, protective treatments, and analysis and measurements.

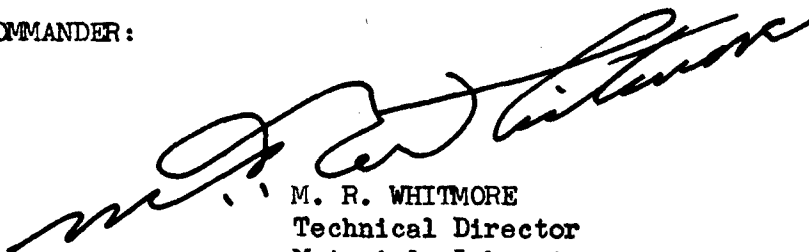
Also listed are seventeen (17) reports issued during 1 June 1951 - 30 June 1953, which were not included in WADC Technical Report 53-373.

As a final summary, a numerical index of all the technical reports issued during the period August 1928 - June 1954 is provided.

## PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



M. R. WHITMORE  
Technical Director  
Materials Laboratory  
Directorate of Research

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ACCESSORIES, AIRCRAFT

TR 53-472 (Unc1)

March 1954

SUBJECT: LOW-TEMPERATURE OPERATION OF AIRCRAFT ACCESSORIES  
INVESTIGATOR: Edward A. Baniak  
CONTRACT: AF 33(038)-15495  
CONTRACTOR: The Texas Company  
ABSTRACT: In order to investigate the performance of greases at low temperatures in actual aircraft accessory equipment, a project was set up which included the testing of five greases in five pieces of accessory equipment. Tests on each of the greases were conducted at room temperature, -65° and -100°F. Of the five greases tested, Greases A, B and C were MIL-G-3278 specification greases, Grease D was a MIL-G-7421 (USAF) Specification grease and Grease E was a high-temperature grease which does not meet any military specification.

From the results of this investigation, the following conclusions were drawn: (1) There was as much variation in the low temperature power requirements of the accessory units of the same design as there were variations attributable to the performance of similar greases. (2) When lubricated with MIL-G-3278 or MIL-G-7421 greases, the power available was sufficient for operating at low temperatures in all motorized equipment except for Item (6), Antenna Assembly. (3) There was no definite correlation between the results of the low temperature torque tests (carried out in No. 204 ball bearings) and the results of the tests on actual aircraft accessory equipment. (4) Grease D was superior to all other greases. (5) Greases A and B were approximately equivalent and slightly superior to Grease C. (6) Grease C was definitely superior to Grease E.

ADHESIVES, STRUCTURAL

TR 53-126 (Unc1)

September 1953

SUBJECT: ELEVATED TEMPERATURE-RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS  
INVESTIGATOR: M. Naps  
CONTRACT: AF 33(600)-6514  
CONTRACTOR: Shell Development Company  
ABSTRACT: A metal-to-metal adhesive which is useful at temperatures up to 500°F has been developed. The adhesive, designated as Formulation No. 422, is a one-package system composed of EPON 1001 resin, a liquid phenolic resin, Plyophen 5023, and dicyandiamide as the curing agent. Aluminum dust is used as the reinforcing filler. The adhesive is cured at contact pressure and at elevated temperature (330°F).

Bonds to aluminum from adhesive Formulation No. 422 have a shear strength of 2100 psi at room temperature and 1400 psi at 500°F. After 200 hours aging at 500°F adhesive shear strength is mediocre (ca 200 psi). Bond strength is, however, 1000 psi after approximately 70 hours aging at 500°F. Aging the adhesive bonds for 200 hours at 400°F reduces the shear strength (at 400°F) from 1750 psi to 1340 psi. Cycling the adhesive bonds between room temperature and elevated temperatures (up to 500°F) for fifty times has had no apparent effect upon the bond strength.

Adhesive Formulation No. 422 is used as a pliable tape, preferably supported on a glass fabric carrier. The adhesive must be stored under refrigeration; storage life at 40°F is about one month.

Systematic variation of the concentration of the components of the adhesive led to the development of the following formula (parts by wt): 33 EPON 1001 + 67 Plyphen 5023 + 100 aluminum dust + 6 dicyandiamide. Higher EPON 1001 resin content decreased hot strength; higher phenolic content increased brittleness and decreased thermal resistance upon aging. Either lower amounts of filler or curing without dicyandiamide reduced adhesive shear strength, especially at room temperature.

The conclusions summarized above represent the status of the work at this writing, but since the work is continuing these conclusions are only tentative. The adhesive 422 is considered an experimental product, and further work is indicated before the adhesive becomes a commercial product.

TR 54-88 (Uncl)

April 1954

SUBJECT: NONDESTRUCTIVE TESTING OF METAL-TO-METAL STRUCTURAL ADHESIVE BONDS  
INVESTIGATOR: B. A. Kulp, M. E. Greenstreet and J. H. Cahn  
CONTRACT: AF 18(600)-124  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: A theoretical study of electrical characteristics of metal to metal adhesive bonded joints, with emphasis toward developing a satisfactory non-destructive test to determine quality of the bond, is presented.

To supplement the theoretical study, three types of experimental tests were conducted on adhesive bonded joints fabricated under close laboratory control. These tests were:

1. sonic - employing a "sonizon", a commercial type instrument.

2. capacitance - using an impedance bridge at 100 kilocycles.
3. d.c. resistance - using a standard ohmmeter.

The results indicate that more extensive research on electrical characteristics of adhesive bonded joints must be conducted to establish definite correlation to bond quality.

#### ALLOYS, FERROUS

TR 53-254, Part 1 (Uncl)

June 1954

SUBJECT: SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT-TREATED TO HIGH STRENGTH LEVELS  
INVESTIGATOR: George Sachs and Walter Beck  
CONTRACT: AF 33(616)-392  
CONTRACTOR: Syracuse University  
ABSTRACT: In Part 1 data on hydrogen embrittlement of high-strength steels are presented and analyzed. Many failures in aircraft reported for steel parts having a strength above 200,000 psi and in addition, many failures of steel bolts heat treated to a strength considerably below 200,000 psi were found to be associated with changes in the basic mechanical characteristics of the steel caused by cadmium plating or chromium plating. Heat treating or baking applied to relieve this hydrogen embrittlement were frequently only partly effective.

Hydrogen in steel is highly mobile and this explains the unusual mechanical features of hydrogen embrittlement and the conflicting results of the many tests which were applied to disclose and measure hydrogen embrittlement. The normal strength of a high-strength steel is found to be rarely affected while its ductility may be greatly reduced. Consequently the strength of test specimens and parts which depends upon ductility may also be much reduced by hydrogen embrittlement. Furthermore, these effects are pronounced at low rates of straining or sustained loading but probably of no significance on impact loading.

The magnitude of hydrogen embrittlement also depends greatly upon numerous mechanical, chemical and electrochemical factors which are encountered in the making, shaping, heat treating, and finishing of aircraft parts.

TR 53-256 (Uncl)

October 1953

SUBJECT: FATIGUE OF ALLOY STEELS AT HIGH-STRESS LEVELS  
INVESTIGATOR: Ture T. Oberg and Edward J. Ward  
ABSTRACT: Several alloy steels used in aircraft have been investigated with the purpose of determining the fatigue strengths at stresses

higher than are found in the stress-cycle fatigue diagrams as usually determined. The stress-cycle diagrams have been extended down to about 1000 cycles and the effects of internal heating at high stresses shown.

ALLOYS, HIGH TEMPERATURE

TR 53-272 (Uncl)

August 1953

SUBJECT: CHROMIUM-BASE ALLOYS  
INVESTIGATOR: Richard G. Nelson and Harry G. Anderson  
CONTRACT: AF 33(038)-50-1084 E  
CONTRACTOR: U. S. Bureau of Mines  
ABSTRACT: Binary alloys of 60Cr-40Fe with oxygen analyses of 0.01 to 0.03 show elongations of 15 and 17% in the hot worked and heat treated conditions. Similar alloys with 0.2% oxygen show up to 14% elongation in the heat treated conditions with little or no elongation in the hot worked state. Alloys with 70% chromium show evidence of ductility. Addition of deoxidizers lowers the ductility by causing intergranular failure. Creep rupture values are given for 60 and 70% chromium balance iron alloys. High chromium alloys can be worked at relatively low temperatures.

TR 53-274 (Uncl)

December 1953

SUBJECT: CASTING AND FORGING TURBINE BUCKET ALLOYS  
INVESTIGATOR: R. K. Pitler and W. W. Dyrkacz  
CONTRACT: AF 18(600)-149  
CONTRACTOR: Allegheny Ludlum Steel Corporation  
ABSTRACT: Wrought nickel-base alloys and chromium-manganese austenitic steels, as well as cast iron-nickel-cobalt-chromium alloys were investigated with an aim toward lowering the strategic alloy content of materials for gas turbine service at temperatures of 1200° to 1600°F. The best nickel-base alloys contained around 10% cobalt and over 5% molybdenum in addition to the age-hardening agents, titanium and aluminum. The austenitic steels, containing 17% manganese and 12% chromium showed promise for service at 1200°F. The addition of boron contributed materially to the high-temperature properties of the cast alloys.

TR 53-277, Part 1 (Uncl)

November 1953

SUBJECT: HIGH-TEMPERATURE PROPERTIES OF FOUR LOW-ALLOY STEELS FOR JET-ENGINE TURBINE WHEELS  
INVESTIGATOR: Arthur Zonder, Adron I. Rush and James W. Freeman  
CONTRACT: AF 33(038)-13496  
CONTRACTOR: University of Michigan  
ABSTRACT: Properties at 1000°, 1100° and 1200°F are reported for

jet-engine turbine wheels made from four low-alloy hardenable steels. The steels were SAE 4340, 1.25 Cr-Mo-Si-V ("17-22A"S), 3 Cr-Mo-W-V (H-40), and 12 Cr-Mo-W-V (C-422). The wheels were contour forged from commercial heats. Wheels of each alloy were heat treated by normalizing, oil quenching, and by an interrupted quench. All were tempered to an aim hardness of 280 to 320 Brinell.

There was very little difference in strength between the alloys for the wheels with the best heat treatment at short time periods. The 4340 steel fell off rapidly in strength with time. The "17-22A"S steel had the highest strength at 1000°F, but fell off with time at 1100° and 1200°F. The H-40 and C-422 steels maintained their strength better with time and temperature. Heat treatments which produced intermediate transformation products in the structures of the steels resulted in highest strength, except for C-422 alloy. The latter 12 Cr alloy could not be heat treated to such structures. The treatments producing the best properties were normalizing for 4340 steel, oil quenching for "17-22A"S steel, oil quenching for H-40, and oil quenching for C-422 alloy.

TR 53-336, Part 1 (Uncl)

May 1954

SUBJECT: THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. PART 1 - INTERMITTENT STRESSING

INVESTIGATOR: Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman and John E. Dorn

CONTRACT: AF 33(038)-11502

CONTRACTOR: University of California

ABSTRACT: Constant and intermittent load creep tests were performed on clad aluminum alloys 75S-T6 and 24S-T3 at 300°, 450° and 600°F, and on cold rolled titanium at 400°F. Constant load creep tests were also conducted on annealed titanium Ti-75-A at 400°F.

Two groups of intermittent load tests were performed, each having a full cycle of two hours. One group was maintained under load for one hour out of every two, and the second for one and one-half hours out of every two.

An empirical method for estimating and correlating creep curves for various stresses at constant temperature has been determined and shown to be applicable to both static and intermittent creep of a number of metals and alloys.

On the basis of total test time, constant load creep was more rapid than creep under either of the two load cycles.

The two alternate types of interrupted loading cycles used in the present investigation gave practically the same creep strain versus net time under load curves. Undoubtedly other types of on and off loading periods would have revealed differences in the creep behavior.

TR 53-451 (Uncl)

January 1954

SUBJECT: THE DEVELOPMENT OF A FORGEABLE HIGH-STRENGTH, HIGH-TEMPERATURE, CHROMIUM-RICH, CHROMIUM-IRON ALLOY

INVESTIGATOR: D. P. Moon, I. A. Blank and A. M. Hall

CONTRACT: AF 33(616)-222

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: The development of a forgeable high-strength, high-temperature, chromium-rich, chromium-iron alloy by Battelle Memorial Institute is described. Experimental alloys were produced by induction melting 4- and 12-pound charges of commercially available melting stock, casting into rammed zirconite molds, and forging to 5/8-inch-square bars for testing. Stress-rupture properties of alloys containing approximately 70Cr, 30Fe, 9Mo, 2 to 3Ti, and up to 1/2Al (parts by weight) were excellent, but room-temperature ductility was poor. Some ductility was obtained after heat treatment in warm-rolled strip of the compositions 55Cr, 45Fe, 1Ti and 60Cr, 40Fe, and 1Ti. It is concluded that the 70Cr-30Fe-base alloy containing about 9Mo, 2 to 3Ti, and up to 1/2Al shows promise as a material of construction for turbine buckets in turbojet aircraft.

TR 54-40 (Uncl)

April 1954

SUBJECT: THE EFFECT OF GRAIN SIZE ON THE FATIGUE AND CREEP PROPERTIES OF STAINLESS STEEL AND INCONEL AT ELEVATED TEMPERATURES

INVESTIGATOR: A. T. Robinson and J. E. Dorn

CONTRACT: AF 33(038)-22608

CONTRACTOR: University of California

ABSTRACT: This investigation was undertaken to evaluate the effect of grain size, as produced by annealing, on the high temperature creep and fatigue properties of inconel and an 18-8 stainless steel. In order to isolate the effects of grain size and annealing from other metallurgical effects, an attempt was made to select materials which were not particularly prone to extraneous changes such as precipitation or spheroidization of intermediate phases. But, in order to estimate the practical utility of practicing annealing for grain size control of high temperature alloys, the two representative alloys inconel and type 304 extra low carbon 18-8 stainless steel were chosen. Unfortunately both of these materials were found to exhibit structural changes during annealing which might have affected their creep and fatigue properties. The results indicate that to clearly delineate the effect of grain size on the properties of metals it will be necessary to use metals in which all auxiliary microstructural changes are absent.

ALLOYS, NONFERROUS, ALUMINUM

TR 53-151, Supplement 1 (Uncl)

October 1953

SUBJECT: STUDY OF HARD COATING FOR ALUMINUM ALLOYS  
INVESTIGATOR: F. J. Gillig  
CONTRACT: AF 18(600)-98  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.  
ABSTRACT: The program for the study of the effects of hard oxide coatings (produced by the MHC Process) on the properties of aluminum and its alloys was extended in order to provide additional data. The corrosion resistance in three environments was evaluated up to 11 months. The abrasion resistance showed another small decrease with the five-month additional exposure to atmospheric and high humidity conditions. Two treatments that were given the coatings on 61S and 75S alloys appear to alleviate the drastic reduction in fatigue strength brought about by the coatings. Attempts at retaining the abrasion resistance in a humid atmosphere were only moderately successful. Oil was found to have a detrimental effect on the resistance to an erosion type of wear.

TR 53-527 (Uncl)

February 1954

SUBJECT: INVESTIGATION OF THE DEGASSING EFFECT OF ULTRASONICS ON ALUMINUM ALLOY  
INVESTIGATOR: J. Byron Jones and John G. Thomas  
CONTRACT: AF 18(600)-32  
CONTRACTOR: Aeroprojects, Incorporated  
ABSTRACT: In this study an attempt has been made to degas molten aluminum by causing gas to collect and rise to the surface from the nodes of sound waves introduced into a melt. The apparatus for effecting this phenomena has been successfully built and demonstrated. In this work certain problems remain unanswered however, since the degassing was erratic and accompanied by serious contamination of the melt by erosion of the transducer coupler material. Efforts to solve the problems will be made in future work.

ALLOYS, NONFERROUS, MAGNESIUM

TR 53-113 (Uncl)

October 1953

SUBJECT: MAGNESIUM ALLOY RESEARCH  
INVESTIGATOR: A. Jones and R. R. Nash  
CONTRACT: W33-038-ac-22542  
CONTRACTOR: Rensselaer Polytechnic Institute  
ABSTRACT: A tentative constitutional diagram is presented for the magnesium-rich portion of the magnesium-thorium alloy system. This diagram shows limited solid solubility and a eutectic. The maximum solid

solubility is estimated as 10 percent at the eutectic temperature of 1105°F. The eutectic composition contains about 35 percent thorium and the eutectic structure consists of an alpha solid solution and an intermediate phase containing about 55 percent thorium.

Tentative diagrams are given to show the effect of zirconium on the phase boundaries for magnesium-zinc-zirconium alloys containing four and six percent zinc, respectively. These diagrams are from data obtained by thermal analysis, microscopic examination and, to a limited extent, x-ray diffraction of a small number of alloys. The liquidus temperature increases slightly with an increase of zirconium in the four percent zinc alloys but remains nearly constant in the six percent zinc alloys. Very little data are given for the solidus and solvus temperatures.

Mechanical property data are given showing a good combination of strength and elongation in magnesium-1% zinc-0.4% cerium, magnesium-1% zinc-0.5% calcium, magnesium-1% zinc-0.4% cerium-0.001% lithium and magnesium-1% zinc-0.4% cerium-0.005% calcium. Cerium refines the grain structure and introduces a second constituent. The best properties are associated with a fine equiaxed grain structure.

Conditions and procedures are given for the controlled solidification method which produced magnesium and binary magnesium alloy single crystals with a nominal diameter of 1/2 inch and a length of about 8 inches. These crystals contain up to 5 percent indium, 5 percent cadmium, 0.5 percent zinc, 0.5 percent silver and 0.3 percent aluminum, respectively. Procedures are given for stressing the crystals in tension to establish the strengthening effects of the individual solutes. Work is reported up to the actual experimental stressing of the single crystals.

TR 53-181 (Uncl)

August 1953

SUBJECT: FATIGUE PROPERTIES OF EXTRUDED MAGNESIUM ALLOY ZK 60  
UNDER VARIOUS COMBINATIONS OF ALTERNATING AND MEAN  
AXIAL STRESSES

INVESTIGATOR: A. A. Blatherwick and B. J. Lazan

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: Axial-stress fatigue tests were conducted on specimens of extruded magnesium alloy ZK60A-T5 under five selected ratios of alternating to mean stress. The data thus obtained are presented in the form of S-N diagrams, one curve for each stress ratio. The effects of varying the combinations of alternating and mean stresses are demonstrated by a series of stress-range diagrams. Three types of specimens (one un-notched and two notched) provide data for analysis of the fatigue strength reduction due to stress concentration. These data are illustrated by two

types of charts showing the fatigue strength-reduction factor as a function of stress ratio, life, and stress magnitude.

TR 54-83, Part 1 (Uncl)

June 1954

SUBJECT: EXPERIMENTAL MAGNESIUM ALLOYS. PART 1 - FURTHER DEVELOPMENT OF PELLET FABRICATED MAGNESIUM ALLOYS  
 INVESTIGATOR: H. A. Johnson, 1st Lt, USAF  
 CONTRACT: AF (600)-19147  
 ABSTRACT: The effect of melt and Mg-Al pellet additions on the properties of Mg-Zn-Zr pellet and/or ingot fabrications was investigated in an attempt to develop higher strength sheet and extrusion alloys. The best combination of properties of Mg-Zn-Zr + Mg-Al pellet extrusions is obtained with ZK60; addition of Mg-Al decreases the sensitivity of the extrusion to annealing but does not improve the overall properties of the base alloy. Certain melt additions, however, do result in significant improvements in the properties of Mg-Zn-Zr. In extrusions, QZ66 containing 2% MM offers much higher strength, particularly CYS, than ZK60, while in sheet ZK40 containing 1%MM and 1%Th has properties vastly superior to AZ31. The following shows this comparison on the basis of laboratory work.

Extrusions* - T5					Sheet* - H26				
1000 psi					1000 psi				
ALLOY	%E	TYS	CYS	TS	ALLOY	%E	TYS	CYS	TS
ZK60	9	45	38	53	AZ31	8	36	31	43
QZ66-2MM	6	49	50	58	ZK40-1Th	8	40	40	49

\*Fabricated from ingots

Comparative properties of extrusions made from pellets and ingot clearly indicate a very marked superiority of the pellet fabrications. Melt additions, however, have smaller effects in pellet than in ingot fabrications.

TR 54-83, Part 2 (Uncl)

June 1954

SUBJECT: EXPERIMENTAL MAGNESIUM ALLOYS. PART 2 - WROUGHT ALLOY SURVEY OF MINOR ADDITIONS TO SELECTED Mg-BASE ALLOYS  
 INVESTIGATOR: H. A. Johnson, 1st Lt, USAF  
 CONTRACT: AF (600)-19147  
 ABSTRACT: The effect of 1% additions of Ba, Cb, Cr, Hg, Li, MM, Mo, Pd, Se, Ta, Te, Th, Ti, Tl, V, and W on the mechanical properties, workability, formability, corrosion resistance, and microstructure of Mg-3Zn and Mg-5Al was investigated. Th greatly improves the strength of Mg-3Zn in both extrusions and rolled strip without an appreciable

loss of toughness (NBE) as illustrated below:

<u>Alloy</u>	<u>Extrusion-T5</u>					<u>Rolled Strip-H24</u>			
	<u>1000 psi</u>					<u>1000 psi</u>			
	<u>%E</u>	<u>TYS</u>	<u>CYS</u>	<u>TS</u>	<u>NBE</u>	<u>%E</u>	<u>TYS</u>	<u>CYS</u>	<u>TS</u>
Mg-3Zn	17	22	12	35	25	13	24	18	33
Mg-3Zn-1Th	14	31	20	40	23	16	32	24	39

Smaller strength increases are obtained by the addition of Li, Ba, and Pd to both base alloys and with Hg and Cr in Mg-3Zn. Sizeable strength increases, through the addition of Th, Li, Pd, or Ba, are achieved only with an appreciable loss in workability, toughness, formability, and/or corrosion resistance. The other additions, Cb, MM, Mo, Se, Ta, Te, Ti, Tl, V, and W, have negligible effects.

TR 54-83, Part 3 (Uncl)

June 1954

SUBJECT: EXPERIMENTAL MAGNESIUM ALLOYS. PART 3 - THERMAL AND ELECTRICAL PROPERTIES OF MAGNESIUM BASE ALLOYS  
 INVESTIGATOR: H. A. Johnson, 1st Lt, USAF  
 CONTRACT: AF (600)-19147  
 ABSTRACT: The effect of crystal orientation on the electrical resistivity of high-purity magnesium was determined. The results, at 24C, may be represented by the equation:

$$\rho(\phi) = 4.60 - 0.75 \cos^2 \phi,$$

where  $\phi$  is the angle between the hexagonal axis and the direction of current flow.

The effect of temperature on the electrical resistivity for varying orientations was investigated, with temperature coefficients of .00390 and .00408/C obtained perpendicular and parallel to the hexagonal axis respectively for the temperature range of 24 to 200°C.

Extruded high-purity magnesium and ZK60A-T5 alloy were stressed in tension to various levels and the effect of this pre-stressing operation on the electrical resistivity recorded. There was no significant change in electrical resistivity at the levels investigated for either alloy.

Apparatus for the measurement of thermal conductivity, electrical conductivity and linear thermal expansion have been purchased or designed but are still in either the construction or calibration stage.

The advantages and disadvantages of electric resistance strain gages as a method of measuring the coefficient of linear thermal

expansion are discussed.

TR 54-83, Part 4 (Uncl)

June 1954

SUBJECT: EXPERIMENTAL MAGNESIUM ALLOYS. PART 4 - EVALUATION OF HK31  
INVESTIGATOR: H. A. Johnson, 1st Lt, USAF  
CONTRACT: AF (600)-19147  
ABSTRACT: This phase of work on contract no. AF (600)-19147 includes the testing and evaluation of production rolled HK31XA sheet. The results are preliminary insofar as the overall production development of this alloy is concerned but do indicate the good combination of room and elevated temperature properties previously obtained in the laboratory can be produced under plant-scale rolling conditions. Continued development work and customer evaluation is warranted.

TR 54-83, Part 5 (Uncl)

June 1954

SUBJECT: EXPERIMENTAL MAGNESIUM ALLOYS. PART 5 - WELDING TESTS ON HK31A SHEET  
INVESTIGATOR: H. A. Johnson, 1st Lt, USAF  
CONTRACT: AF (600)-19147  
ABSTRACT: The arc, gas and spot welding characteristics of HK31A sheet and plate were investigated. The alloy was very weldable by the arc and spot methods but difficulty was encountered in gas welding. Heavy plate, multiple pass arc welds were subject to oxide inclusions, the cause of which has not been definitely established. Weld strengths could be improved by aging heat treatments. The welds were subject to germination when solution heat treated at the recommended 1050°F temperature. The short time high temperature properties of the welds were not significantly different from the unwelded material. Spot welding techniques and properties appeared to be similar to those obtained on AZ31A sheet. Suggestions for continuing the evaluation of the welding characteristics of HK31A were included.

ALLOYS, NONFERROUS, TITANIUM

TR 52-335 (Uncl)

November 1953

SUBJECT: TITANIUM PHASE DIAGRAMS  
INVESTIGATOR: Harold D. Kessler, William Rostoker and Robert J. Van Thyne  
CONTRACT: AF 33(038)-8708  
CONTRACTOR: Armour Research Foundation Illinois Institute of Technology  
ABSTRACT: Using high purity arc melted alloys and micrographic analysis of annealed samples as the principle method of investigation,

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titanium-rich, partial phase diagrams were determined for the following systems;

**Ti-Cr-Fe:** The titanium-rich portion of the diagram was studied in detail to 70 weight % titanium. The isotherm at 800°C was determined for the section bounded by Ti,  $\text{TiFe}_2$ , and  $\text{TiCr}_2$ . The solubility of chromium and iron in alpha titanium is less than 1% total alloy content. There is a continuous space of ternary beta solid solution between the Ti-Cr and Ti-Fe systems. A ternary eutectoid,  $\beta \rightleftharpoons \alpha + \text{TiCr}_2 + \text{TiFe}$ , occurs at approximately 8% Cr-13% Fe and about 540°C. The beta phase of alloys lying on the low titanium side of a tie line between 7% chromium and 4% iron is retained upon water quenching from the beta space. Hardness data are presented, illustrating the marked effect of heat treatment.

**Ti-Al-O and Ti-Al-N:** Titanium-rich corners of the systems from 0 to 10% aluminum and 0 to 1% oxygen or nitrogen were investigated. As aluminum, oxygen and nitrogen are alpha-stabilizers, the ternary alpha spaces extend to temperatures well above the transformation temperature of titanium (885°C). Nitrogen is more effective than oxygen in raising the  $\beta/\alpha + \beta$  space boundary of the Ti-Al system. Upon water quenching the alloys, the beta phase transforms to alpha prime. Oxygen and nitrogen additions increase the hardness of the Ti-Al alloys.

**Ti-Al-C:** The phase diagram was determined using alloys containing 0 to 10% aluminum and 0 to 1% carbon. A study of several as-cast carbon master alloys permitted an outline of the Ti-C diagram to be constructed. A high melting compound,  $\text{TiC}$ , appears to exist over a range of compositions. A eutectic occurs at about 30% carbon between  $\text{TiC}$  and carbon.

Aluminum raises the temperature of the peritectoid reaction of the Ti-C system,  $\beta \rightleftharpoons \alpha + \text{TiC}$  (920°C). The maximum solubility of carbon in alpha titanium is increased by aluminum additions from about 0.5% in the binary Ti-C system to 1% at 10% aluminum. Aluminum and carbon increase the hardness of the alpha solid solution.

**Ternary Oxide Phases:** A family of ternary oxide phases isomorphous with  $\text{Fe}_3\text{W}_3\text{C}$  was discovered in which titanium was associated with one of the elements of the first transition series. The phase relationships between these ternary phases were investigated for the Ti-Cr-O, Ti-Fe-O and Ti-Ni-O systems. An isothermal section for the Ti-Mo-O system was constructed.

TR 53-109, Part 1 (Uncl)

July 1953

SUBJECT: THE TENTATIVE TITANIUM-SILVER BINARY SYSTEM  
INVESTIGATOR: Henry K. Adenstedt and William R. Freeman, Jr.,  
1st Lt, USAF

**ABSTRACT:** Silver is a beta former when alloyed in small amounts with titanium. There is a large range of solubility of silver in alpha titanium and it reaches a maximum of  $9 \pm 1$  wt.% silver at  $1560 \pm 7^\circ\text{F}$ , at which temperature an eutectoid reaction,  $\beta \rightarrow \alpha + \text{TiAg}$ , occurs at  $17 \pm 0.5$  wt.% silver. The beta solid solubility increases to  $30.5 \pm 1$  wt.% silver at  $1903 \pm 5^\circ\text{F}$ .

Contrary to general opinion, the existence of the compound TiAg (69.25 wt.% Ag) has been established. It is formed at  $1903 \pm 5^\circ\text{F}$  by the following peritectic reaction:  $\beta + \text{Liq.} \rightleftharpoons \text{TiAg}$ . The composition limits of the compound have, as yet, not been determined; however, the compound field appears to be narrow from preliminary observations.

Titanium is soluble in silver in an amount of less than 0.5 wt.% as two phases are present in 99.5 wt.% silver as-cast alloys.

TR 53-177 (Uncl)

August 1953

**SUBJECT:** TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION  
**INVESTIGATOR:** W. F. Carew, F. A. Crossley, H. D. Kessler and M. Hansen  
**CONTRACT:** AF 33(038)-22806  
**CONTRACTOR:** Armour Research Foundation of Illinois Institute of Technology

**ABSTRACT:** Results on the following phases of the investigation are presented: (1) homogeneity of the double melted eight-pound ingots, (2) density determinations, (3) structural stability studies, (4) creep rupture testing, (5) room and elevated temperature tensile testing and (6) an evaluation of the effect of exposure to air at elevated temperatures on stress rupture properties. Alloys for which experimental data are reported are: 2% Al, 4% Al, 6% Al, 8% Al, 6% Al-1% Si, 8% Al-1% Si, 8% Al-4% Mo, 6% Al-1, 3 and 5% Ta, 8% Al-1, 3 and 5% Ta and 30% Mo.

Homogeneity of the eight-pound ingots was generally good. The structures developed by the heat treatments used appeared to be stable under the conditions of creep rupture testing. At  $550^\circ\text{C}$  for a service life of approximately 1000 hours, the results show that the alloys 6% Al-1% Si, 8% Al-1% Si and 8% Al-4% Mo are superior in creep resistance to stainless steel, Type 410 (12.5% Cr-bal. Fe). Also, at  $425^\circ\text{C}$  and probably  $550^\circ\text{C}$  for 1000 hours service life, the 6% Al alloy is shown to have superior creep resistance to Type 410. Alloys 6% Al-1, 3, 5% Ta and 8% Al-1, 3, 5% Ta offer little, if any, advantage in rupture strength over their 6 and 8% Al binary counterparts at  $550^\circ\text{C}$  and are inferior to the 6% Al-1% Si alloy. Exposure to air at elevated temperatures for 100 hours appears to have little or no effect on the creep rupture properties of the titanium-aluminum-base alloys.

May 1954

SUBJECT: STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET

INVESTIGATOR: Herbert M. Meyer and William Rostoker

CONTRACT: AF 33(616)-206

CONTRACTOR: Armour Research Foundation

ABSTRACT: The reactions of three basic categories of titanium alloys to heliarc welding have been studied. The report covers work performed during the period 15 June 1952 to 15 July 1953. Studies included the influence of preheat-and-postheat-treatments on the mechanical properties of welded specimens. Mechanical properties included bend ductility, tensile strength and elongation, and hardness. Microstructures were examined extensively with a view to recording their relationships to weld behavior. A large number of alloys proved extremely brittle in the as-welded state. In almost every case, a large measure of ductility could be restored by postheat-treatment. Preheat-treatment did not generally improve the ductility of the as-welded state. No single postheat-treatment procedure was found applicable to all alloys. A number of commercial alloys was examined. In particular, the RC-130A alloy was studied in some detail, and brittle welds could be rendered ductile by postheat-treatment.

#### ANALYSIS AND MEASUREMENT

TR 53-201, Part 1

October 1953

SUBJECT: THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 1 - THE HEAT CAPACITY OF LITHIUM FLUORIDE FROM 0°C TO 900°C

INVESTIGATOR: Thomas B. Douglas and James L. Dever

CONTRACT: AF (33-616)52-10

CONTRACTOR: U. S. Department of Commerce National Bureau of Standards

ABSTRACT: By use of an ice calorimeter and a furnace containing a silver core, the relative enthalpy of lithium fluoride was measured from 0° to 900°C. These measurements, at nine temperatures for the solid and three for the liquid, possessed a probable error of the mean of 0.03% on the average, and led to a heat of fusion of 1043.6(±5) absolute joules per gram. The melting point was measured and found to be 848°C(±1°). After the sample had been frozen rapidly, the mean heat capacity between 0° and 550° was found to be lower by 0.2%. The derived heat capacity values of the solid are believed to be accurate in general to within ±0.75%, and the mean heat capacity of the liquid between 848° and 900°C, to ±1.5%. The heat capacity - temperature curve of the solid is in good agreement with values recently reported in the literature for the range 19° to 272°K (-254° to -1°C).

August 1953

SUBJECT: THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 2 - THE HEAT CAPACITY OF ANHYDROUS SODIUM HYDROXIDE FROM 0° to 700°C.

INVESTIGATOR: Thomas B. Douglas and James L. Dever

CONTRACT: AF 33(616)-52-10

CONTRACTOR: National Bureau of Standards

ABSTRACT: Samples of sodium hydroxide whose purity analyses indicated to be approximately 99.3% were sealed in pure silver. Cryoscopic measurements indicated a freezing point of 317° and a transition temperature of 293°C. Using a "drop" method and a Bunsen ice calorimeter, the enthalpy change between 0° and each of eleven temperatures up to 700°C was measured. With reasonable assumptions, the thermal measurements near the freezing point were used to correct for the impurity, giving for pure sodium hydroxide a melting point of 319°C and heats of fusion and transition of 159 and 158 absolute joules per gram, respectively. The heat capacity derived for the liquid is estimated to be correct within two percent. Values of heat capacity, entropy, relative enthalpy, and relative free energy are tabulated between 298° and 1000°K.

October 1953

SUBJECT: THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 3 - THE HEAT CAPACITY OF MOLYBDENUM DISILICIDE FROM 0° TO 900°C

INVESTIGATOR: Thomas B. Douglas and William M. Logan

CONTRACT: AF 33(616)-52-10

CONTRACTOR: National Bureau of Standards

ABSTRACT: The enthalpy relative to 0°C of a sample of molybdenum disilicide ( $\text{MoSi}_2$ ) was measured at 100-degree intervals up to 900°C, using a nichrome-V container for the sample, a silver-core furnace, and a precision ice calorimeter. Corrections were applied for the impurities (approximately 2%) reported to be in the sample. The equation

$$C_p = 0.1019 + 4.32(10^{-5})t - 1.70(10^{-8})t^2,$$

giving the heat capacity in calories per gram - deg C at  $t^\circ\text{C}$  between 0° and 900°C, represents the unsmoothed corrected values derived from the data with an average deviation of 0.8%. This equation is believed to have an absolute accuracy within  $\pm 3\%$  between 100° and 800°. Values of the enthalpy, heat capacity, and relative entropy derived from this equation are formulated and tabulated at round temperatures.

November 1953

SUBJECT: DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 1 - DENSITY OF SODIUM AND SODIUM HYDROXIDE

INVESTIGATOR: M. Nishibayashi  
 CONTRACT: AF 33(616)-9  
 CONTRACTOR: University of Cincinnati  
 ABSTRACT: A densitometer to be used for liquids at temperatures up to ca. 1000°C in an inert atmosphere was constructed from grade "A" nickel. In the construction of this densitometer, the hydrostatic weighing method of Kohlrausch was selected as the method most suitable for the determination of density under the conditions imposed. This method utilizes the buoyancy principle of Archimedes. A plummet is suspended from one arm of an analytical balance and is immersed in the sample contained in the densitometer tube. The apparent loss in weight of the plummet upon immersion is measured by a chainomatic balance. The entire apparatus is gas-tight; observations and manipulations of the balance are made through gas-tight seals. With this apparatus the density of a material can be measured from its melting point to ca. 1000°C using a single sample of the material.

The density of sodium and sodium hydroxide was measured from 254° to 860°C and from 447° to 736°C respectively. The following equations relating density to temperature were derived from the experimental data.

$$d_{Na} = (0.9378 \pm 0.0086) - (0.000268 \pm 0.000018)(t - 97.5^\circ)$$

$$d_{NaOH} = (1.753 \pm 0.002) - (0.000565 \pm 0.000008)(t - 318.4^\circ)$$

Using these equations, the average deviations between the observed and the calculated values of density were found to be 0.93% for sodium and 0.23% for sodium hydroxide.

TR 53-378 (Uncl)

January 1954

SUBJECT: THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET  
 INVESTIGATOR: George A. Dalin, Ph.D. and Robert W. Freedman, Ph.D.  
 CONTRACT: AF 33(616)-111  
 CONTRACTOR: Balco Research Laboratories  
 ABSTRACT: Methods are discussed for the preparation of transparent, conductive films on both acrylic and glass surfaces. The theory of the structure of such films is presented as well as a description of techniques involved in the preparation. Techniques described are sputtering of metallic cadmium under conditions such as to form the oxide, and the preparation of thin continuous films of silica and titania.

TR 53-465 (Uncl)

October 1953

SUBJECT: PREPARATION OF HOMOGENEOUS OXYGEN BEARING IODIDE TITANIUM SPECIMENS

INVESTIGATOR: Rodger D. Veneklasen  
CONTRACT: AF 33(616)-2041  
CONTRACTOR: National Research Corporation  
ABSTRACT: Five 5-pound ingots have been alloyed, melted and fabricated to produce the homogeneous oxygen bearing iodide purity titanium specified in the contract. Chemical analyses for interstitial elements, carbon, nitrogen and tungsten have been made on the ingots. Oxygen and hydrogen analyses have been made by vacuum fusion analytical methods developed in the laboratories of National Research Corporation. Analytical results are close to intended oxygen compositions and show the individual samples to be uniform and homogeneous within the limits of accuracy of the analytical methods. These analyses further show the interstitial elements to be lower than the limits set in the contract. The oxygen alloys were to have nominal compositions of 0.1, 0.2, 0.5, 1.0% with one additional ingot serving as a blank.

It is felt that the procedure used for obtaining uniform oxygen content is reliable and accurate and it will be applied to other research work now in progress and requiring uniform additions of oxygen to Ti and Ti alloys. It is anticipated that further research applications will be forthcoming requiring controlled additions of oxygen, nitrogen or carbon to metals requiring cold mold melting.

#### BIOCHEMISTRY

TR 6518, Part 4 (Uncl)

January 1954

SUBJECT: FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COMPOUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 4 - FLUORINATED PHENOLS, BENZYL ALCOHOL, AND BIPHENYLS  
INVESTIGATOR: Leo R. Tehon  
CONTRACT: AF 33(038)-10897  
CONTRACTOR: Illinois State Natural History Survey  
ABSTRACT: The fungistatic activities of eight aromatic fluorine compounds were investigated. The group was heterogeneous, comprising one chlorinated phenol, two nitrophenols, one benzyl alcohol, three biphenyls, and one biphenyl sulfide.

Potencies of each compound toward four fungi deleterious to cotton fabric were determined first by the agar dilution plate method. Of the more potent compounds, further tests were made to gauge their effectiveness in protecting cotton parachute webbing thread.

Least potent of the compounds was the benzyl alcohol. Most potent with a range of effectiveness of from 8.8 to 22.5 parts per million for the test fungi, was 4-fluoro-2-chlorophenol. The biphenyls, although only moderately potent, offered such advantages as lack of color.

a solid state, and high melting point. Introduction of sulfur in the biphenyl linkage appreciably increased the potency of one compound.

In protection tests with parachute webbing thread compound content in treated thread of from 1.6 to 3.7 per cent, depending on the compound, was sufficient to prevent diminution of tensile strength in the presence of an actively growing fungus.

TR 54-30 (Uncl)

February 1954

SUBJECT: THE INVESTIGATION OF FUNGICIDES FOR LEATHER  
INVESTIGATOR: Martin A. Townsend, 1st Lt, USAF and Paul A. Albert, Capt, USAF

ABSTRACT: Forty-two experimental formulations containing fungicidal chemicals have been evaluated as protective treatments against mildew on leather. Twenty-one were formulations containing orthophenylphenol, eleven formulations contained fluorinated compounds, three formulations contained trichlorophenyl acetate, three formulations contained paranitrophenol, two formulations contained parachlorometaxylenol, and the remaining two formulations contained di-lauryl dimethyl ammonium bromide and 2,2 dihydroxy -5,5' dichlorodiphenyl methane, as the active fungicides. To determine the fungistatic effectiveness of each treatment, at least one of two methods was employed; the Petri plate method (mycelial mat), and the mixed spore suspension method. With the Petri plate method, only the fungus Aspergillus niger was used. With the mixed spore suspension method fourteen fungi were used.

All of the treatments inhibited the growth of fungi to some degree, with the exception of the formulations containing parachlorometaxylenol, 2,2' dihydroxy -5,5' - dichlorodiphenyl methane, and di-lauryldimethyl ammonium bromide as the active fungicides.

In addition, most of the treatments which inhibited the growth of fungi were no more corrosive than the untreated leather, when in contact with cadmium plated steel, clad 24S-T3 aluminum alloy and brass.

Toxicity evaluations of orthophenylphenol and the fluorinated benzene derivatives show that these fungicides are non-toxic under the conditions studied.

#### CERAMICS

TR 53-91 (Uncl)

July 1953

SUBJECT: INVESTIGATION OF REFRACTORIES SUITABLE FOR MELTING  
TITANIUM AND ITS ALLOYS

INVESTIGATOR: Marvin Eisenberg and James Stavrolakis  
CONTRACT: AF 33(038)-23280  
CONTRACTOR: Armour Research Foundation Illinois Institute of Technology

ABSTRACT: Theoretical analysis of the published data pertaining to simple and complex fluorides suggested that it would be possible to produce certain fluoride and oxyfluoride complexes with melting points high enough to render them infusible at the temperature of molten titanium. Because of their potentially high chemical stability and the noncontaminating nature of the products of reaction between fluorides and titanium, fluorides seem particularly desirable as possible container materials for molten titanium.

Attempts were made to prepare several synthetic oxyfluoride minerals, as well as a number of new oxyfluoride compounds. Though it was possible to produce highly refractory fluoride ceramics, success was not attained in producing the exact compound desired. Nor was the product completely stable within the required temperature range. An analysis of the results suggests that the optimum fabrication technique was not achieved.

Under the handicap of pronounced instability, a number of the complex fluoride refractories produced were nevertheless superior to zirconia in resistance to molten titanium. However, a product suitable for industrial application has not yet been produced.

TR 54-1 (Uncl)

May 1954

SUBJECT: PHYSICAL CHARACTERISTICS IN TITANIUM-CARBIDE-BASE CERMETS AT ELEVATED TEMPERATURES

INVESTIGATOR: Friedrich P. Knudsen, R. Eugene Moreland and Roman F. Geller

CONTRACT: AF 33(616)-52-2

CONTRACTOR: National Bureau of Standards

ABSTRACT: The project's principal purpose was to determine, at temperatures of 1200°F to 2000°F, the creep behavior and strength of cermets which may find application in the high temperature areas of aircraft.

Initially a series of bending tests were made on variations of Kennametal's composition K151A. The variations contained from 5% to 30% nickel bonding. The 5%, 15% and 20% compositions were recommended, on the basis of their creep resistance and high strength, for further long-term tensile creep tests.

A limited number of tensile creep tests were made on Kennametal's compositions K151A-20% nickel, K151A-25% nickel, K152A, K162B and on Firth Sterling's composition, FS-27. The cermet K162B was

deemed the most promising of the tested compositions, with respect to the properties of creep resistance and high strength. Metallographic examinations were made of all the tensile tested specimens and, in general, the test strains had been too small to cause any noticeable deformation or changes.

Thermal expansion determinations were made on Kenna-metal's compositions K138, K138A, K151 and K151A. The coefficients of expansion between room temperature and 1205°C ranged from  $8.1 \times 10^{-6}$  to  $8.6 \times 10^{-6}$ .

TR 54-13 (Uncl)

February 1954

SUBJECT: INVESTIGATION OF THE EFFECT OF RAW MATERIAL PRODUCTION VARIABLES ON THE PHYSICAL AND CHEMICAL PROPERTIES OF CARBIDES, NITRIDES, AND BORIDES

INVESTIGATOR: Herman Blumenthal

CONTRACT: AF 33(616)-89

CONTRACTOR: American Electro Metal Corporation

ABSTRACT: The purpose of this project is to investigate the effect of raw material production variables on the physical and chemical properties of carbides, nitrides, and borides. In the investigations carried out so far, this aim has been narrowed down to the study of titanium carbide.

Various titanium carbides, produced commercially by as many different procedures as possible, have been purchased and are being used for this investigation. The various materials have been analyzed spectrographically, chemically and by x-ray diffraction to determine the amounts and kinds of impurities peculiar to the production technique employed. Changes of chemical composition during ball milling, hot pressing and other processing steps leading to the production of solid pieces have been followed by the same analytical techniques as well as metallographic studies.

The question of the effect of the presence of impurities has been approached from two sides, namely (1) by removing and (2) by adding impurities. Some impurities, such as iron, graphite and oxide films, have been partly removed by purification treatments like leaching with acids and flotation. Other impurities, such as TiO, TiN and graphite, have been added to pure TiC.

The effect of various ball milling media on the chemical composition of the milled product has been studied.

In the production of unbonded TiC bars, hot pressing has been used exclusively, while Ni-bonded bars have been produced (1)

by hot pressing and (2) by cold pressing followed by sintering either under a protective atmosphere or in vacuum.

Infiltration experiments have been conducted as an attempt to develop a test procedure for the evaluation of quality of titanium carbide powders.

Hot pressed unbonded bars have been tested for the following properties: maximum density obtainable, ability to infiltrate, and electrical resistivity.

It has been found that the chemical composition of TiC produced by different procedures varies as to combined and free carbon, oxygen and nitrogen content as well as metallic impurities.

The amount and kind of impurities present influence maximum density obtainable by hot pressing unbonded bars, and their ability to infiltrate.

The amount of iron and oxygen picked up during ball milling in a steel mill varies greatly with the ball milling medium.

Ni-bonded bars of high density could be produced so far only by hot pressing. Cold pressing followed by sintering in hydrogen or in vacuum resulted in bars of low density.

The ability of a porous bar to infiltrate depends on its density, impurity content, and particle size of the original powder.

TR 54-27 (Uncl)

May 1954

SUBJECT: THE EFFECTS OF PRE-COMPRESSION ON THE THERMAL SHOCK RESISTANCE OF PURE OXIDE CERAMICS  
INVESTIGATOR: Darwin Marshall and Dwight G. Bennett  
CONTRACT: AF 33(616)-87  
CONTRACTOR: University of Illinois  
ABSTRACT: A combination thermal cycling and compressive loading apparatus was constructed for investigating the effects of pre-compression on the thermal shock resistance of a pure oxide ceramic body.

It was found that selective compressive loads applied in the direction of the long axis of a rectangular bar composed of alpha alumina effectively increased its thermal shock resistance. This was indicated by a progressive increase in the average modulus of rupture of each of several groups of bars; each group being uniformly thermal shocked at a compressive load of 1000,

3500 and 7000 psi, respectively. It was also found that about one half of the total improvement was obtained with the initial load of 1000 psi.

The sonic technique was used as a method of detecting flaws in thermal shocked but unloaded specimens. When specimens were thermal shocked in compression, however, the sonic method, as used in this investigation, was unable to indicate the presence of flaws.

#### CLEANING

TR 53-147 (Uncl)

November 1953

SUBJECT: NONTOXIC SOLVENT FOR CLEANING AIRCRAFT ENGINES AT EXTREMELY LOW TEMPERATURES  
INVESTIGATOR: Bernard Berkeley and Isidore Shafiroff  
CONTRACT: AF 33(616)-36  
CONTRACTOR: Foster D. Snell, Incorporated  
ABSTRACT: Two aircraft engine degreasers consisting essentially of methylene chloride and aliphatic petroleum hydrocarbon were recommended on the basis of laboratory evaluations as being suitable for use in arctic operations. Toxicity, flash point, vapor pressure and stability data on known low melting point solvents were compiled and screened for the purpose of selecting basic and additive ingredients of the degreasers. A correlation between evaporation rates at -65°F and 77°F was established for pure compounds. The results of previous government field tests dealing with the evaluation of low temperature engine degreasers were proved to be misleading and incorrect. Synthetic soiling mixtures comparable to actual engine soil were created for this project on the basis of field reports and laboratory tests with engine soil. The recommended degreasers were evaluated for detergency properties, flash point, corrosive action on metals, effect on accessory equipment, toxicity and evaporation rates.

#### CORROSION

TR 6519, Part 3 (Uncl)

January 1954

SUBJECT: MATERIALS FOR HANDLING FUMING NITRIC ACID AND PROPERTIES OF FUMING NITRIC ACID WITH REFERENCE TO ITS THERMAL STABILITY  
INVESTIGATOR: Mars G. Fontana  
CONTRACT: AF 33(038)-10381  
CONTRACTOR: The Ohio State University Research Foundation  
ABSTRACT: Polarization and corrosion studies were made on galvanic couple systems in WFNA. Aluminum was found to be anodic to stain-

less steel in the aluminum-stainless steel couple; this resulted in rapid corrosion of the aluminum and almost complete protection of the stainless steel. A theory on the mechanism of anodic corrosion of aluminum is proposed. The effect of water additions to WFNA on the corrosion of couples is discussed. Corrosion studies on welded Type 321 stainless steel show this material to be susceptible to knife-line attack similar to that reported for Type 347 stainless steel. An apparatus for studying the erosion-corrosion of metals and alloys in FNA is described.

A series of measurements of the rate of decomposition and the equilibrium decomposition pressure of pure nitric acid were carried out in glass tubes. The effect of the ratio of vapor volume to total volume of the sample,  $V_g/V$ , on the rate and equilibrium pressure was studied in detail at 76°C. The reversibility of the decomposition reaction when carried out at constant volume conditions was satisfactorily demonstrated. Evidence is given to show that the equilibrium pressure at a given temperature and  $V_g/V$  ratio, is the same whether the equilibrium is approached from a higher or lower temperature. A study of the kinetics of the reaction at 76°C indicates that the reaction occurs in the liquid phase and is one of the first order.

TR 53-16, Part 2 (Uncl)

March 1954

SUBJECT: CORROSION PREVENTIVE ADDITIVES  
INVESTIGATOR: E. J. Schwoegler and L. U. Berman  
CONTRACT: AF 33(038)-9202  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: This project was undertaken to develop new corrosion inhibitors that may supplement or replace petroleum sulfonates. Wright Air Development Center Technical Report 53-16 Pt 1 described a number of types of organic compounds which had been effectively screened as corrosion inhibitors by the Static Water Drop Test. This evaluation study has been continued with major emphasis on the type of compounds which previously were shown to be effective inhibitors, and the minimum effective concentration was determined. With certain amine salt inhibitors a study was made to determine the effect of heating at 150°C for three hours on corrosion inhibition.

A large number of organic compounds were screened by the use of a Galvanic Specimen Test also in order to discover inhibitors that might be effective for inhibiting corrosion in the bi-metallic system, 52100 steel-Muntz metal brass. In the course of this investigation, conditions of temperature, humidity, etc. were studied to arrive at conditions best suited for screening these compounds.

During the course of this program, the initial studies were made on the mechanism of corrosion inhibition of 1020 steel. In the elucidation of the mechanism of corrosion inhibition, attempts were made to relate effectiveness to the structural and functional group characteristics of the molecules studied. The compound types studied included morpholine derivatives, aliphatic amines and amine salts of various carboxylic acids in both AN-O-6a and MIL-L-6085A base oils.

Many organic compounds were synthesized to assist in the development of new inhibitors and in the elucidation of the mechanism of corrosion inhibition. These included morpholine and ethylenediamine derivatives, amine salts of mono- and dicarboxylic acids, fatty acid derivatives and substituted glyoxalidines.

An infrared study on 2- and 1,2-substituted glyoxalidines was made in an effort to arrive at an effective method of verifying these structures. This was essential since certain glyoxalidines were found to have potential inhibiting properties sufficient to warrant further investigation of this type of compound.

#### CRITERIA, DESIGN

TR 5662, Part 5 (Uncl)

December 1953

SUBJECT: INVESTIGATION OF MECHANICAL PROPERTIES AND PHYSICAL METALLURGY OF AIRCRAFT ALLOYS AT VERY LOW TEMPERATURES. PART 5 - MECHANICAL PROPERTIES OF METALS AND A PLASTIC LAMINATE AT LOW TEMPERATURES

INVESTIGATOR: M. G. Fontana, S. M. Bishop and J. W. Spretnak

CONTRACT: AF 33(038)-15698

CONTRACTOR: The Ohio State University Research Foundation

ABSTRACT: The materials and tests covered in this supplementary report are as follows:

- (1) Tensile and notched fatigue tests at  $-253^{\circ}\text{C}$  on 24S-T4, 75S-T6, NE(SAE) 8630 steel, titanium, and 18-8 stainless steel type 304.
- (2) Tensile compression, and fatigue tests at room temperature and  $-196^{\circ}\text{C}$  on a glass-fabric-polyester resin laminate.
- (3) Hardness, tensile and impact tests of titanium at room temperature and  $-78^{\circ}\text{C}$ .

- (4) Hardness and impact tests of a Cr-Ni-Cu steel at room temperature,  $-78^{\circ}\text{C}$ ,  $-127^{\circ}\text{C}$ ,  $-196^{\circ}\text{C}$ , and  $-253^{\circ}\text{C}$ .
- (5) Dilatometric measurements over the range room temperature to  $-253^{\circ}\text{C}$  on 24S-T4, 75S-T6, NE(SAE) 8630 steel, titanium, and 18-8 stainless steel.
- (6) Thermal cycling tests of the glass fabric polyester resin laminate consisting of cooling to  $-196^{\circ}\text{C}$  and warming to room temperature.

The results may be summarized as follows:

- (1) In tension, all the materials are notch sensitive at  $-253^{\circ}\text{C}$ ; the ductility of the NE 8630 steel is greatly reduced; the other materials retain some ductility at  $-253^{\circ}\text{C}$ . At  $-253^{\circ}\text{C}$ , all the materials tested show an improvement in fatigue resistance at low stresses. The resistance to overstressing is reduced.
- (2) Both the compressive and tensile strengths of the polyester resin laminate are improved at  $-196^{\circ}\text{C}$ . Some increase in fatigue strength results when this material is cooled to  $-196^{\circ}\text{C}$ .
- (3) Titanium increases in hardness and tensile strength at  $-78^{\circ}\text{C}$ , but its impact resistance is lowered.
- (4) The hardness of the Cr-Ni-Cu steel is increased as temperature is lowered. This material undergoes a gradual transition from ductile to brittle impact fracture as the temperature is lowered to  $-253^{\circ}\text{C}$ .
- (5) The aluminum alloys have the highest coefficients of expansion at low temperatures. Titanium shows a low expansion coefficient.
- (6) The glass fabric polyester resin laminate is not damaged by repeated thermal cycling between room temperature and  $-196^{\circ}\text{C}$ .

TR 6517, Part 2 (Uncl)

April 1954

SUBJECT: DETERMINATION OF PHYSICAL PROPERTIES OF FERROUS AND

NONFERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED  
TEMPERATURES

INVESTIGATOR: D. D. Doerr  
CONTRACT: AF 33(038)-8681  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: In order to establish design criteria, the compressive, bearing and shear properties have been determined for (1) XA78S-T6 aluminum alloy sheet (clad), and FS-1a magnesium alloy at room temperature and at elevated temperatures ranging from 212° to 600°F, for exposure periods between 0.5 and 1,000 hours, (2) Heat Treat Nos. 1 and 2 of SAE 8630 alloy steel, SAE 4130 alloy steel, annealed stainless steel, and half-hard stainless steel, at room temperature and at several elevated temperatures between 400° and 1200°F for exposure periods ranging from 0.5 to 100 hours. These properties have been compared with the tensile data obtained under corresponding conditions in an attempt to establish whether a correlation exists which would permit prediction of the compressive, bearing and shear characteristics from the tensile properties of the individual material at room and elevated temperatures.

The test specimens, equipment, and procedures are described in detail. The test results are presented in the form of tables and curves to illustrate the effect of temperature and exposure time on the physical properties of the various materials under investigation.

TR 6517, Part 3 (Uncl)

June 1954

SUBJECT: DETERMINATION OF PHYSICAL PROPERTIES OF FERROUS AND  
NONFERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED  
TEMPERATURES  
INVESTIGATOR: Donald E. Miller  
CONTRACT: AF 33(038)-8681  
CONTRACTOR: Armour Research Foundation Illinois Institute of  
Technology  
ABSTRACT: In order to establish important design criteria, tensile, compressive, bearing, and shear properties have been determined for the following materials and conditions: (1) 14S-T6 aluminum alloy sheet (clad) at room temperature and at elevated temperatures ranging from 200° to 600°F, for exposure periods between 0.5 and 1000 hours; (2) 24S-T81 and 24S-T86 aluminum alloy sheet (clad) at room temperature and at 200°, 300°, and 400°F for exposure periods between 0.5 and 1000 hours; (3) FS1-H24 magnesium alloy sheet at 200°F, for exposure periods of 0.5 and 1000 hours; (4) 75S-T6 aluminum alloy sheet (clad) at 200°F, for exposure periods between 0.5 and 1000 hours; (5) cold rolled titanium and annealed titanium at 200°F, for exposure periods of 0.5 and 1000 hours; and (6) RC-130-A titanium alloy at room temperature and at temperatures ranging from 300° to 800°F, for ex-

posure periods of 0.5, 100, and 1000 hours. A comparison was made between the tensile data and the data on other properties in an attempt to formulate a method for estimating all other elevated temperature properties from a knowledge of tensile elevated temperature properties and room temperature values of other properties. The conclusion was reached that the various properties are not related in a simple, consistent manner.

Test specimens, equipment, and procedures are described in detail. Test results are presented in the form of tables and curves to illustrate the effect of temperature and exposure time on the mechanical properties of the various materials under investigation.

TR 52-251, Part 2 (Uncl)

May 1954

SUBJECT: INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM COLUMNS AT ELEVATED TEMPERATURES. PART 2 - STABILITY PROBLEMS

INVESTIGATOR: R. L. Carlson and G. K. Manning

CONTRACT: AF 33(038)-9542

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A method for estimating allowable load capacities of columns subject to creep is presented. The method, which utilizes approximate stress distributions derived from isochronous-stress-strain curves to estimate column load capacities, is shown to be conservative for the time for which the estimate is made.

An application of the method is made to test data on as-received and on stabilized 24S-T4 aluminum alloy. A comparison of the computed column capacities with experimental capacities indicates that the method is satisfactory for estimating the decrease in capacity with increasing time.

Easily obtained, time-dependent tangent-modulus loads are discussed. They are interpreted as being approximations to allowable load-capacity estimates. A limited application is made to test data, and the results appear promising. It is concluded that if certain limitations are recognized, the method may prove to be useful because of its simplicity.

A presentation of the results of an experimental investigation of the effects of column imperfection and column-material variation is made. It is found that column-capacity variations of the order of 10 per cent can result from column-imperfection differences and column-material variation.

The results of an experimental study of the variation of column capacity with temperature of exposure are presented.

They indicate that column efficiency, as measured by decrease in capacity, can be acceptable for very short times at higher temperatures. The efficiency at these higher temperatures falls rapidly, however, with increasing time.

TR 53-71 (Uncl)

September 1953

SUBJECT: EFFECT OF STRAIN RATE ON THE MECHANICAL PROPERTIES OF TITANIUM-BASE MATERIALS  
INVESTIGATOR: D. R. Luster, W. W. Wentz and J. P. Catlin  
CONTRACT: AF 33(038)-21912  
CONTRACTOR: Rem-Cru Titanium, Inc.  
ABSTRACT: The results of this investigation show that at intermediate rates of strain, high purity titanium is not significantly more sensitive to strain rate changes than are some other metals. Nitrogen additions, however, increase the rate sensitivity markedly so that the presence of nitrogen in all commercial titanium-base materials is the probable reason for their high rate sensitivity at intermediate and low strain rates.

All of the representative types of titanium alloys were investigated. The substitutional all-alpha alloy showed the most desirable strength -vs- strain rate characteristics. This alloy type exhibited the lowest rate sensitivity at the slow testing speeds thus promising good long-term strength, and also exhibited a relatively rapid strengthening with high rate of strain, thus promising excellent impact resistance. The high impact resistance of the all-alpha alloy was confirmed by Charpy V-notch impact tests.

Analysis of the test data and examination of microstructures suggests that all titanium-base material undergo a basically similar change in mode of deformation with changing strain rate and temperature. It is proposed that this change is from slip and/or twinning at high strain rates and/or low temperatures to grain boundary microflow at low strain rates and/or high temperatures. Some evidence is given although further confirmation is required.

The effect of strengthening the base material by cold working or alloying appears to be to alter the range of conditions over which this changing mode of deformation takes place. Cold working and alloying also superimpose strain aging, recovery, and transformation effects which further alter the individual characteristics of the various representative titanium-base materials investigated.

TR 53-300 (Uncl)

November 1953

SUBJECT: MATERIAL PROPERTY DESIGN CRITERIA FOR LARGE MAGNESIUM ALLOY CASTINGS

INVESTIGATOR: Keith F. Finlay  
CONTRACT: AF 33(600)-8496  
CONTRACTOR: Northrop Aircraft, Inc.  
ABSTRACT: A study was made of the correlation between casting size, metallurgical quality as determined by radiographic classification, and mechanical properties of large magnesium alloy castings.

Twelve 40-inch panels and one 16-foot wing section were cast. Standard separately cast test bars were produced as controls for each casting. The 40-inch and 16-foot castings were radiographed in their entirety to determine the sections which represented a range of metallurgical imperfections. Representative imperfect sections were removed and machined into test coupons. The coupons were in turn radiographed and classified. Tensile tests, compression tests, shear tests, and strain measurements were carried out on the coupons, and statistical analysis was conducted on the resulting data.

Data, graphs showing the results of statistical analysis, and conclusions and recommendations are presented in this report. In addition, photomicrographs representing typical radiographic classifications are included. A supplement contains glossy prints of the radiographs for all test specimens.

TR 53-510, Part 1 (Uncl)

March 1954

SUBJECT: DYNAMIC CREEP, STRESS-RUPTURE, AND FATIGUE PROPERTIES OF 24S-T4 ALUMINUM AT ELEVATED TEMPERATURES.  
PART 1 - UNNOTCHED SPECIMENS  
INVESTIGATOR: Fred W. DeMoney and Benjamin J. Lazan  
CONTRACT: AF 33(038)-20840  
CONTRACTOR: University of Minnesota  
ABSTRACT: Fatigue, stress-rupture, and creep data obtained under various combinations of mean and alternating axial stress are presented and discussed for rolled aluminum alloy 24S-T4 at 300° and 500°F. The data are presented as S-N curves and stress range diagrams to show the effect of temperature, alternating-to-mean load ratio, and stress magnitude on the fatigue, stress-rupture, and creep properties.

The effect of temperature and alternating-to-mean ratio on the characteristics of the creep curve is discussed on the basis of "static" and "dynamic" types.

The role of both creep and fatigue as factors in rupture is discussed with particular reference to temperature and alternating-to-mean ratio.

ELECTRODEPOSITION

TR 5692, Supplement 4 (Uncl)

July 1953

SUBJECT: AN INVESTIGATION OF ELECTRODEPOSITED ALLOYS FOR PROTECTION OF STEEL AIRCRAFT PARTS  
INVESTIGATOR: Arch B. Tripler, Jr., Glen Fuller and Dr. Charles L. Faust  
CONTRACT: AF 33(038)-8750  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Pure-manganese, manganese-zinc-alloy, and zinc-tin-alloy coatings on steel were exposed for 21 months at the Battelle North Florida Research Station. Panels coated with zinc, cadmium, and chromated zinc were exposed concurrently as standards.

The pure-manganese and manganese-zinc coatings were inferior to the zinc and cadmium standards.

The zinc-tin-alloy coatings of 0.3 mil thickness and greater protected the underlying steel as well as the zinc and cadmium standards.

TR 53-162, Part 1 (Uncl)

September 1953

SUBJECT: ELECTRODEPOSITION OF TITANIUM  
INVESTIGATOR: Albert W. Schlechten, Martin E. Straumanis and C. Burroughs Gill  
CONTRACT: AF 33(616)-75  
CONTRACTOR: Missouri School of Mines and Metallurgy  
ABSTRACT: The high resistance of titanium to corrosion, particularly by sea water or nitric acid, makes it very desirable to plate other metals with a protective coating of titanium. Many attempts by many workers have been made to electrodeposit titanium either to form a coating or as a means of producing the metal but little success has been reported. This report describes a large number of experiments using aqueous and fused salt baths. It is doubtful if any true electrodeposits of titanium were obtained, but a procedure is described which will yield a thin but coherent and corrosion resistant titanium coating. Extensive data are also reported on the hydrogen overvoltage on titanium in aqueous electrolytes.

TR 53-218 (Uncl)

November 1953

SUBJECT: DEVELOPMENT OF A NONDESTRUCTIVE TEST FOR EVALUATION OF ADHESION OF ELECTRODEPOSITS ON STEEL AS IN SILVER-PLATED AIRCRAFT BEARINGS  
INVESTIGATOR: Arch L. Walters and Samuel A. Wenk  
CONTRACT: AF 18(600)-124

CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report surveys the nondestructive test methods currently being used for the inspection of silver-plated aircraft bearings. Details of the two principal test methods are given.

The selection of the test method depends on whether a copper or nickel strike is applied to the bearing shell before silver plating. The shot peening test, wherein the surface of the silver plate is lightly shot peened under controlled conditions is principally used on bearings having a copper strike. The nickel strike bearings are tested by heating the bearing to 950°F, followed by rough boring, or machining to size and then X-raying the bearing surface.

Shot peening will produce wrinkles in poorly bonded areas. The heating method produces blisters, which are readily revealed by X-ray examination following machining.

In addition, the report contains "Proposed Amendments to Technical Order No. 24-5-1 on Electrodeposition of Silver Plate". It suggests changes in the Technical Order for the purpose of improving the procedure for silver plating and reducing the occurrence of poorly adherent silver plate at Air Force depots overhauling aircraft engines.

TR 53-271 (Uncl)

July 1953

SUBJECT: DEVELOPMENT OF A SUBSTITUTE OR IMPROVEMENT OF CHROMIUM ELECTRODEPOSITS  
INVESTIGATOR: Jesse E. Stareck, Edgar J. Seyb, Jr. and Angelo C. Tulumello  
CONTRACT: AF 33(616)-234  
CONTRACTOR: United Chromium, Incorporated  
ABSTRACT: The fatigue limit of heat-treated steel was studied with chromium deposits from various solutions with varying plating conditions. Baseline information was obtained from the conventional sulfate bath and the newer high speed CR-110 bath. The fatigue limit was shown to increase with increased fineness of crack pattern in the chromium deposit. By adjusting the plating conditions to give more filled-in cracks the fatigue limit was materially increased. Deposits with very few crack-lines showed extremely low fatigue limits.

Baking chromium deposits to eliminate hydrogen embrittlement drastically lowered the fatigue limit in most cases. Certain types of deposits possessing oxide inclusions gave higher rather than lower results after baking. A copper strike between chromium and steel was shown to largely prevent embrittlement without changing the fatigue properties.

The data is taken to show that residual tensile stress in electrolytic chromium is the primary mechanism for the lowering of the fatigue limit in chromium plated steel.

TR 53-317 (Uncl)

December 1953

SUBJECT: ELECTRODEPOSITION OF TITANIUM AND ZIRCONIUM  
INVESTIGATOR: Robert M. Creamer, David H. Chambers and Charles E. White  
CONTRACT: AF 33(038)-50-1085  
CONTRACTOR: U. S. Bureau of Mines  
ABSTRACT: Aqueous, non-aqueous, and fused electrolytes have been investigated in an attempt to electrodeposit titanium and zirconium. Aqueous and non-aqueous electrolytes have been found capable only of coplating small percentages of titanium along with another more easily plated metal such as tin, cadmium, or zinc. Fused alkali borate baths were found to give non-adherent, spotty cathode deposits of a titanium boride. Fused halide electrolytes gave powdered metal deposits and a potassium chloride-lithium chloride-titanium (III) chloride electrolyte gave weighable plates on iron, nickel, and copper cathodes. Notes on some of the chemical aspects of the investigation are included.

#### FATIGUE

TR 53-122 (Uncl)

August 1953

SUBJECT: REVIEW OF PREVIOUS WORK ON SHORT-TIME TESTS FOR PREDICTING FATIGUE PROPERTIES OF MATERIALS  
INVESTIGATOR: Franz H. Vitovec and Benjamin J. Lazan  
CONTRACT: AF 33(038)-20840  
CONTRACTOR: University of Minnesota  
ABSTRACT: Experimental observations relating to the fatigue process and theories of fatigue are briefly reviewed. Short-time fatigue testing methods are systematically reviewed and critically discussed. The classification of the methods is based on the relationship of fatigue properties to static properties, to stress-strain characteristics under reversed stress, and to other physical properties. Other methods discussed involve assumptions regarding the shape of the S-N curve. Also tests utilizing special loading conditions are reviewed. Advantages and applicability of the different methods are discussed.

TR 53-167 (Uncl)

September 1953

SUBJECT: THE EFFECT OF SPECIMEN SURFACE AS A DISCONTINUITY IN FATIGUE PHENOMENA  
INVESTIGATOR: F. H. Vitovec  
CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota  
ABSTRACT: Experimental observations relating to the fatigue process and theories of fatigue are discussed. A picture of the mechanism of fatigue considering all experimental observations is developed. The influence of the free surface of the specimen on the strength of surface grains is discussed. Considering the effect of the free specimen surface, the influence of size of specimen, shape of cross section, and the effect of notches on fatigue strength are explained. The effect of sharp notches in a material is shown in relationship to the propagation of fatigue cracks.

TR 53-184 (Uncl)

November 1953

SUBJECT: FATIGUE PREDICTION BY MEANS OF THE CYCLOGRAPH  
INVESTIGATOR: R. L. Cavanagh  
CONTRACT: AF 33(038)-19151  
CONTRACTOR: Ontario Research Foundation  
ABSTRACT: Tests were conducted on SAE 4340 samples in different heat treated conditions to determine whether changes in magnetic and electrical properties at low field strengths could be correlated with fatigue damage. To indicate the changes in magnetic and electrical properties, the Cyclograph was used. This is a high frequency, non-destructive, magnetic test instrument. Some tests were also conducted to evaluate the application of the method for a short fatigue test to obtain the endurance limit.

Test equipment, specimens and procedures are described in detail. Test results are presented in the form of tables and curves. The static bend test in combination with the fatigue test seems to be the best and most convenient test of those investigated to attempt to determine fatigue damage. It can generally be concluded that it is possible to determine whether a steel part has been loaded above or below the endurance limit while it is in service and that by application of low static loads in the same sense as the fatigue load it appears that the extent of fatigue damage can be approximated.

TR 53-393, Part 1 (Uncl)

December 1953

SUBJECT: DEVELOPMENT OF A FATIGUE DAMAGE INDICATOR: PART 1 - PRELIMINARY INVESTIGATION  
INVESTIGATOR: Darnley M. Howard  
CONTRACT: AF 33(038)-51-4061  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: This report describes an investigation that was conducted in an attempt to develop a fatigue damage indicator which can be used in aircraft to warn of impending fatigue failure. Five

different methods of indicating fatigue damage were investigated, four were subsequently abandoned as all tests on these four methods were unsuccessful. The fifth method investigated is based on Miner's theory of cumulative damage and measures fatigue damage by means of a fine aluminum alloy wire bonded to an aluminum alloy fatigue specimen. The wire is subjected to a known amount of fatigue damage before it is attached to the specimen and is therefore expected to fail in fatigue before the specimen and give warning of impending failure in the specimen. The results of tests on this method indicate that further tests might produce a workable fatigue damage indicator.

TR 53-437 (Uncl)

February 1954

SUBJECT: THE INFLUENCE OF SURFACE TREATMENT ON THE FATIGUE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS

INVESTIGATOR: Lars Thomassen, Maurice J. Sinnott and Albert W. Demmler, Jr.

CONTRACT: AF 33(616)-26

CONTRACTOR: University of Michigan

ABSTRACT: The fatigue properties of commercially pure titanium (Ti-75A) and a titanium alloy (RC-130B), as determined by a rotating beam type of test, have been evaluated as a function of several surface conditions. A shot-peened surface improves the fatigue properties of both alloys although the effect on RC-130B is noticeable only at the higher stress levels. A ground surface on both alloys showed the poorest fatigue properties of any of the surfaces that were prepared by mechanical methods. Four other types of surfaces prepared in different ways such as hand finishing, rough machining, and electro-polishing showed some significant differences in fatigue properties but generally were quite similar. Oxidizing or nitriding the surface of both alloys markedly lowers the fatigue strength.

TR 54-20 (Uncl)

March 1954

SUBJECT: FATIGUE FAILURE UNDER RESONANT VIBRATION CONDITIONS

INVESTIGATOR: B. J. Lazan

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: The nature of resonant vibration and the accompanying amplification of fatigue stress are discussed in relationship to the damping energy absorbed by a vibrating system. The resonance amplification factor is defined as a measure of the severity of a resonant condition. The sources of damping in a vibrating system are discussed and classified according to whether they are external (structural) or internal (material). Data on the

internal damping properties of a variety of structural materials are presented and the generalized behavior is discussed. In cases where internal damping is significant, the importance of both fatigue strength and damping properties of materials as joint criteria for resonant strength is demonstrated and quantitatively expressed. The analyses are made in terms of the resonant strength constant for the material (the material factor) and the volume-stress function of the part (the part factor).

TR 54-64 (Uncl)

March 1954

SUBJECT: FRICTIONAL DAMPING AND RESONANT VIBRATION CHARACTERISTICS OF AN AXIAL SLIP LAP JOINT  
INVESTIGATOR: James H. Klumpp and Benjamin J. Lazan  
CONTRACT: AF 33(038)-20840  
CONTRACTOR: University of Minnesota  
ABSTRACT: A brief summary of the various methods of damping analysis is presented. Data are procured on the frictional properties of mild steel in reciprocating sliding motion. The variation of the kinetic coefficient of friction as a function of normal load, lubrication, and number of cycles of motion is studied. The friction testing apparatus is considered as a vibrating system with Coulomb damping and its frequency response and damping energy are analysed.

#### JOINING

TR 52-313, Part 2 (Uncl)

December 1953

SUBJECT: BRAZING TITANIUM TO TITANIUM AND TO MILD AND STAINLESS STEEL  
INVESTIGATOR: W. J. Lewis, G. E. Faulkner, P. J. Rieppel and C. B. Voldrich  
CONTRACT: AF 33(038)-23338  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: The brazing characteristics of titanium when joined to titanium, mild steel, and stainless steel were studied. Joints between these materials were induction brazed, torch brazed, and furnace brazed with commercial and experimental brazing alloys.

Induction-brazed joints produced higher strengths than furnace- and torch-brazed joints. Joints in titanium were more easily brazed by all methods and using all alloys than those between titanium and mild and stainless steels.

High-strength joints in titanium were obtained using titanium-base experimental brazing alloys, but ductility was poor.

January 1954

SUBJECT: PRODUCTION OF SOUND DUCTILE JOINTS IN MOLYBDENUM  
INVESTIGATOR: M. I. Jacobson, D. C. Martin and C. B. Voldrich  
CONTRACT: AF 33(616)-10  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Methods of welding and brazing molybdenum with the objective of producing sound ductile joints were investigated by Battelle Memorial Institute for the Wright Air Development Center during the period 25 March 1952 to 25 September 1953. The results of tungsten-arc welding tests showed that, although ductile weld metal could be produced, the welded joints were brittle transverse to the direction of welding because of recrystallization in the base metal. Several brazing methods were investigated, with induction brazing in an argon atmosphere appearing to be the most satisfactory from the standpoint of braze quality and practicability. Brazed joints were tested at room temperature and at 1800°F. The brazing alloys that gave the best results at 1800°F were Inconel and Haynes Alloy 25, which produced joints with 100-hour shear strengths of 5000 psi and 4500 psi, respectively.

TR 53-231 (Uncl)

December 1953

SUBJECT: RECRYSTALLIZATION WELDING OF ALUMINUM AIRCRAFT ALLOYS  
INVESTIGATOR: Nickolas A. DeCecco and John M. Parks  
CONTRACT: AF 18(600)-92  
CONTRACTOR: Armour Research Foundation Illinois Institute of Technology  
ABSTRACT: Recrystallization is established as the mechanism for solid phase welding. 2S, 24S, 52S and 61S aluminum alloys were welded by pressing together two cleaned and cold worked surfaces and heating to the recrystallization temperature. The shear strengths of the welded joints are equivalent to that of the annealed metal.

The fundamental forces in operation when making a recrystallization welded joint have been investigated and analyzed. The basic principles and techniques described are applicable to metals other than aluminum and aluminum base alloys.

TR 52-322, Part 2 (Uncl)

February 1954

SUBJECT: CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS  
INVESTIGATOR: A. J. Jacobs, P. J. Rieppel and C. B. Voldrich  
CONTRACT: AF 33(038)-12619  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report discusses an experimental investigation of the causes of cracking in high-strength weld metals. The

research was conducted at Battelle during the third contract period, from August 12, 1952, to August 12, 1953. Specifically, the work included hot-ductility and weld-metal cracking tests on special high-strength steels and a metallographic examination of grain boundaries and inclusions in two of the steels. The results, although in an early stage, indicate that low-sulfur contents are associated with high hot ductility and high resistance to hot cracking. They also suggest a possible relationship between low-melting intergranular films and low hot ductility.

## LEATHER

TR 53-414 (Uncl)

January 1954

SUBJECT: THERMAL AND AREA STABILITY OF LEATHER  
INVESTIGATOR: Herbert C. Hamel, Robert E. Merritt and William T. Roddy  
CONTRACT: AF 33(616-136)  
CONTRACTOR: University of Cincinnati  
ABSTRACT: A comparison of the properties of horsehide versus cowhide leathers was made to determine the feasibility of using cowhide leathers as well as horsehide leathers for glove purposes. The data collected would indicate that cowhide leather would serve the purpose. There was the possibility that the initial run obtained in horsehide glove leather might not be present in the cowhide leather.

Tannage and the process ramifications necessary to each tannage preparation were studied as to their influence on thermal and area stability of leather. For the purpose of study a series of commercial tannages were investigated. The data obtained shows that tannage is a very important factor in preparing fire resistant leather. The data also indicates that the objective of developing a glove leather with higher heat and area stability has been accomplished.

The application of fire retardants to leather was also investigated. The fire retardants selected on the basis of the improvement in fire resistance they imparted to cloth fabrics did not impart good fire resistance to leather. Some of the disadvantages encountered when these materials were applied to leather are given in the report.

## PACKAGING

TR 53-38, Part 1 (Uncl)

January 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 1 - STUDY OF GREASEPROOF BARRIER REQUIREMENTS FOR AN-G-25 GREASE  
INVESTIGATOR: R. S. Kurtenacker  
CONTRACT: AF 18(600)-103  
CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture  
ABSTRACT: Various greaseproof barrier materials were used in this investigation. Twenty-three materials were tested for compliance with Specification JAN-B-121, Barrier-Materials, Greaseproof. Four materials were used as conforming wraps for Method IA-1 packs of bearings, prepared with preservative, Specification AN-C-124, Compound, Soft-Film, Corrosion-Preventive or with grease, Specification AN-G-25, Grease, Low Temperature Aircraft, Lubricating and subjected to exposure tests.

Of the twenty-three materials tested, three met all requirements of Specification JAN-B-121, and five met all requirements except for high acidity.

Tests of Method IA-1 packs indicate that the grease-proofness, as determined by Specification JAN -B-121, is not an indication of satisfactory use with grease, Specification AN-G-25. None of the barrier materials prevented dispersion of the oil base from the grease. Metal surfaces were stained and pitted in all packs subjected to the 8-day cycle plus 90 days' storage, except when material SN was used as the barrier material.

In tests of Method IA-8 packs, the metal-foil pouches appeared to prevent dispersion of the oil base from grease, Specification AN-G-25. Bearings in these pouches were free from stain, while bearings in pouches made of barrier materials, SN, PF, or PL were stained or pitted.

Method IA-8 packs appear suitable for packaging bearings prepared with AN-G-25 grease. It is recommended, however, that additional work be done and that the make-up of the AN-G-25 grease be investigated.

TR 53-38, Part 2 (Uncl)

March 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 2 - INVESTIGATION OF ALUMINUM-FOIL BARRIERS FOR PACKAGING  
INVESTIGATOR: Dr. V. W. Meloche and D. R. Johnson  
CONTRACT: AF 18(600)-103  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: An investigation of the use of aluminum foil as an intimate wrap for bearings coated with either petrolatum-base preservatives or synthetic greases and packaged in water-vaporproof pouches was conducted by Forest Products Laboratory. The aluminum foil in each pack was either oil-free foil, foil with the rolling oil employed in manufacturing the foil, or foil with an excess of rolling oil. The exposure consisted of either three 5-day cyclic tests at temperatures ranging from -65° to +160°F. or this cyclic test plus 60 days at 160°F. and 92 percent relative humidity.

Results indicate that the aluminum foil is subject to pinholding and breaking at the folds, does not retain the grease or preservative with the type of wrap employed, and does not prevent changes in the composition of the synthetic grease. The results also indicate that it is more desirable to employ oil-free foil than the foil with rolling oil present. As a basis for comparison, a polyethylene-coated kraft barrier gave better performance than did the aluminum foil with the synthetic grease.

March 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 3 - COMPATIBILITY OF MIL-G-3278 SYNTHETIC GREASE WITH VARIOUS INTIMATE WRAPS AND AN-B-20 POUCHES (SERIES A)

INVESTIGATOR: M. A. Taras

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: One hundred and twenty-six pouch packages containing the outer ring of standard ball bearings assemblies were subjected to a cyclic exposure to evaluate the compatibility of pouch materials, synthetic greases, and intimate wraps. Various combinations of 2 pouches, 2 greases, and 6 intimate wraps were used in the test packs.

Based on the results of the exposure test, the AN-B-20 pouch materials were about equally effective in protecting the rings from moisture vapor and other agents of corrosion. Both MIL-G-3278 greases caused pouch deterioration; however, one of the greases appeared to be less deleterious than the other. None of the combinations of pouches or intimate wraps prevented hardening of the greases. The polyester film was the only intimate wrap material that produced consistently good results with both greases and pouch materials.

June 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 4 - THE PERFORMANCE OF FIVE METHODS OF PACKAGING BEARINGS IN METAL CONTAINERS (SERIES 1)

INVESTIGATOR: L. O. Anderson

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture

ABSTRACT: This investigation was conducted to determine the relative performance of five methods of packaging bearings as prepared by a roller bearing manufacturer. These cans were subjected to a 3-week cycle with temperature extremes of -65°F. and 160°F., followed by 60 days of exposure at 160°F. and 92 percent relative humidity.

The best results were produced by a preservative of petrolatum and an intimate wrap of neutral kraft waxed paper. The use of silica gel with this combination of preservative and wrap was also satisfactory. The use of AN-C-124, type II preservative resulted in corroded bearings. The use of MIL-G-10924 grease over a petrolatum film with an aluminum wrap did not give fully satisfactory results. The package using dicyclohexylammonium nitrite as a volatile corrosion inhibitor was also unsatisfactory under the conditions of this test.

These results are not fully conclusive, since all of the test specimens were prepared at the adverse condition of 90 percent relative humidity.

February 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 5 - THE PERFORMANCE OF VARIOUS TYPES OF WATER-VAPORPROOF BARRIERS CONTAINING GREASE-COATED PARTS

INVESTIGATOR: M. A. Taras

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture

ABSTRACT: An investigation of the performance of various water-vaporproof barrier materials used as pouches for packaging grease- or preservative-coated parts was conducted by the Forest Products Laboratory.

Five different pouch materials, a synthetic grease, and a petroleum-base preservative were investigated. Twelve packages were made with each pouch material, 6 with synthetic grease and 6 with petroleum-base preservative. Each preservative-or grease-coated ring was wrapped in a greaseproof barrier of polyethylene-kraft. These packages were exposed to a 3-week cycle of temperatures ranging from -65° to 160°F. Following the 3-week exposure period, 1 group of 30 packages was stored for 30 days, and the other group of 30 packages was stored for 60 days at 160°F. and 92 percent relative humidity

Results indicated that pouches of barrier Nos. 60 and 62 do not deteriorate upon exposure with either the grease or preservative used in this experiment. Pouches of barrier No. 63 when used with synthetic grease gave good protection to inner rings of bearings, despite the fact that the pouches deteriorated severely and water droplets were found on the grease. Although two of the barrier materials used for pouches did not deteriorate upon exposure, none of the barrier materials gave good protection to the rings when used with preservative. The exposure condition of 160°F. is too severe for this preservative, since it flows readily from the ferrous parts at this temperature.

March 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 6 - THE PERFORMANCE OF THREE TYPES OF SEALS FOR VARIOUS INTIMATE WRAPS WITH MIL-G-3278 GREASES (SERIES E)

INVESTIGATOR: M. A. Taras

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: A study of the performance of three methods of closing various intimate wraps containing ferrous bearing parts coated with grease complying with Specification MIL-G-3278 was conducted by the Forest Products Laboratory. The three methods for closing the intimate wraps included the drugstore fold, heat sealing, and the use of an aluminum band. All specimens were enclosed in water-vaporproof pouches

complying with Specification MIL-B-131A and were exposed to a 3-week cycle of temperatures ranging from  $-65^{\circ}$  to  $160^{\circ}\text{F}$ . Following the cyclic exposure, the specimens were stored for 60 days at  $160^{\circ}\text{F}$ . and 92 percent relative humidity.

Results indicate that the effectiveness of the three sealing methods investigated is largely dependent upon the compatibility of the intimate wraps and the greases used. When both greases are considered, the three sealing methods appear to be equally effective. Corrosion when present, however, was more severe in packages using the drugstore fold method.

In general, the greases used hardened upon exposure. Nylon, however, was the only intimate wrap that prevented any of the greases from hardening. No reason for this reaction was apparent. The data also indicate that one of the greases was less deleterious to the vinyl coating of the pouch material used in this test than the other.

TR 53-38, Part 7 (Unc1)

February 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 7 - PLASTIC-COATED, ALUMINUM-FOIL BARRIERS AS INTIMATE WRAPS FOR PACKAGING BEARINGS.

INVESTIGATOR: M. A. Taras

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: A study of the performance of plastic-coated aluminum foils as intimate wraps for packaging bearing parts coated with grease or preservative was conducted by the Forest Products Laboratory. Eight different intimate wrap materials, 2 greases, and 2 methods of sealing the intimate wraps were investigated. The specimens were inclosed in water-vaporproof pouches and exposed to a 3-week cycle of temperatures ranging from  $-65^{\circ}$  to  $160^{\circ}\text{F}$ . Following the 3-week exposure period, 1 group of packages was stored for 30 days and another group for 60 days at  $160^{\circ}\text{F}$ . and 92 percent relative humidity.

Results of this study indicated that the heat-seal method of closing laminated plastic-coated aluminum-foil barrier intimate wraps was more effective in regard to corrosion prevention than the drugstore method of closure. The only barrier that offered complete protection to the synthetic grease-coated bearings was a heat-sealed polyethylene-foil-polyethylene-scrim intimate wrap. Vinyl-foil, cellulose acetate-foil and plain aluminum foil gave the poorest performance with the synthetic grease. The cellulose acetate-foil, vinyl-foil, and polyethylene-kraft gave the poorest performance with the AN-C-124 preservative. The plastic coatings on the foil reduced the amount of fracturing of the aluminum foil but did not reduce it sufficiently to keep the grease or preservative within the confines of the intimate wrap.

The synthetic grease had a tendency to deteriorate all the plastic laminated foils severely except the polystyrene-foil combinations.

TR 53-38, Part 8 (Uncl)

January 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 8 - PERFORMANCE OF TIN CANS CONTAINING ANTIFRICTION BEARINGS AND VARIOUS TYPES OF OILS (SERIES P AND Q)  
INVESTIGATOR: L. O. Anderson, M. A. Taras and R. K. Stern  
CONTRACT: AF 18(600)-103  
CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture  
ABSTRACT: An investigation to determine the effectiveness of procoated tin cans for packaging antifriction bearings with various oils was conducted by the U. S. Forest Products Laboratory.

Three types of oils together with three bearing types were investigated. Cans, each containing 1 bearing and a maximum amount of oil, were sealed and subjected to a 3-week cyclic exposure test plus 30, 60, or 120 days of storage at 160°F. and 92 percent relative humidity. In addition, cans containing 2 bearings and a maximum amount of oil were subjected to the 3-week cyclic exposure and 120 days of exposure.

No corrosion was evident on any of the bearings in packages subjected to the 30-day exposure period. Bearings packaged in tin cans with the inhibited oils were in excellent condition after storage for 120 days. Corrosion of a minor extent was present on 50 percent of the bearings packaged in tin cans with the uninhibited oil that were exposed to a 3-week cycle plus 120 days of storage.

No significant difference was evident when 2 bearings, instead of the customary 1, were packaged in a can.

TR 53-38, Part 9 (Uncl)

March 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 9 - PERFORMANCE OF FOUR MIL-G-3278 GREASES WITH VARIOUS GREASE-PROOF BARRIERS IN ANTIFRICTION BEARING-POUCH PACKAGES (SERIES S)  
INVESTIGATOR: Michael A. Taras  
CONTRACT: AF 18(600)-103  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: This experiment to determine the effectiveness of four MIL-G-3278 greases used in combination with various types of intimate wraps in pouch-type packages in preventing corrosion of bearing parts was conducted by the Forest Products Laboratory. Four greases and six intimate wraps were investigated. The specimens were enclosed in water-vaporproof pouches and exposed to a 3-week cycle of temperatures ranging from -65°

to 160°F. Following the 3 weeks of cyclic exposure, one group of packages was stored for 30 days and another group of packages was stored for 60 days at 160°F. and 92 percent relative humidity.

Results of this study showed that various synthetic greases that comply with Specification MIL-G-3278 perform differently at the same storage conditions. It was likely that variation in grease composition was responsible for this reaction.

Nylon (3 mils) was the only barrier that performed satisfactorily with each of the greases.

TR 53-38, Part 10 (Uncl)

February 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 10 - PERFORMANCE OF PLASTIC-COATED (EXTRUDED) ALUMINUM-FOIL BARRIERS AS INTIMATE WRAPS FOR PACKAGING BEARING PARTS COATED WITH SYNTHETIC GREASE

INVESTIGATOR: M. A. Taras and J. O. Bixby

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: This investigation was conducted primarily to determine the performance of aluminum foil with an extruded plastic coating as an intimate wrap in packaging bearing parts coated with MIL-G-3278 synthetic grease. Three hundred pouch-type packages containing the outer rings of ball bearings were prepared with various combinations of grease, coated and uncoated intimate wraps, and methods of closure of the wrap. The test packages were exposed to a 3-week cyclic exposure with temperature extremes of -65°F. and 160°F., followed by 60 days of storage at 160°F. and 92 percent relative humidity.

Severe delamination occurred with polyethylene-foil wraps regardless of the kind of grease or method of closure used. Comparable packages using nylon extruded to foil were not delaminated. Delamination, varying in degrees from none to moderate, was evident in polyethylene-tissue-foil wraps with all greases.

Pinholing (fracturing) of the aluminum foil usually accompanied the use of a fold method of sealing the intimate wrap and was not evident in wraps closed with a heat seal. There was no significant difference, however, in the amount and degree of corrosion in comparable packs that could be attributed to method of closure. Coated foil wraps were usually more effective when the coated surface of the wrap was positioned in contact with the grease-coated bearing part.

Although all four synthetic greases used in this experiment complied with Specification MIL-G-3278, a wide range of effectiveness in corrosion prevention existed among them.

February 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 11 - PERFORMANCE OF PROCOATED BLACK IRON CAN PACKAGES CONTAINING VOLATILE CORROSION INHIBITOR AND ANTIFRICTION BEARINGS (SERIES T)

INVESTIGATOR: V. C. Setterholm

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: This study was initiated to investigate the performance of volatile corrosion inhibitors as protection for two types of anti-friction bearings in procoated black iron cans.

Interior protection was provided by dicyclohexylammonium nitrite-impregnated paper or crystals. Procoated black iron cans were used to contain antifriction bearings with steel or brass retainers.

The packages were exposed at a 3-week cyclic exposure with temperature extremes of  $-65^{\circ}$  to  $160^{\circ}\text{F}$ . One group of bearings with brass retainers were examined after this exposure. Following the 3-week cyclic exposure period, both brass and steel antifriction bearing packages were stored for 60 days at  $160^{\circ}\text{F}$ . and 92 percent relative humidity.

Although the dicyclohexylammonium nitrite was successful in minimizing corrosion of ferrous parts, the tendency of crystals to undergo sublimation resulted in a dense formation of crystals on all surfaces of the bearing. The presence of these crystals on functional surfaces acted to freeze the rolling parts.

The use of MIL-C-10506 procoating material as a protective coating on the black iron cans was unsatisfactory in this test.

February 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 12 - THE EFFECT OF FIVE CLEANING PROCEDURES ON ANTIFRICTION BEARINGS PACKAGED WITH MIL-G-3278 GREASES

INVESTIGATOR: R. A. Aubey

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory

ABSTRACT: A study was made by the Forest Products Laboratory to determine the effectiveness of five cleaning methods in removing contamination and preventing recontamination of antifriction bearings packaged with greases that complied with Specification MIL-G-3278. The grease-coated specimens and a limited number of controls without grease were exposed in two manners. A group of pouch packages was exposed to a 3-week cyclic exposure plus 60 days of storage at  $160^{\circ}\text{F}$ . and 92 percent relative humidity, and another group of grease-coated rollers was placed on open slings and subjected to an exposure of  $80^{\circ}\text{F}$  and 80 percent relative humidity for 88 days.

The results indicated that cleaning Method C-5 of Specification MIL-P-116A, consisting of the use of a specification fingerprint remover followed by a solvent wash, is the most satisfactory. Other cleaning methods were accompanied by staining and discoloration of the specimens. None of the methods prevented recontamination. A thin film of oil did not prevent recontamination.

TR 53-38, Part 13 (Uncl)

January 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 13 - PERFORMANCE OF TIN CANS CONTAINING ANTIFRICTION BEARINGS SUBJECTED TO CYCLIC EXPOSURE AND ROUGH HANDLING (SERIES F-F)

INVESTIGATOR: R. K. Stern

CONTRACT: AF 18(600)-103

ABSTRACT: The principal objective of this study was to determine the performance of procoated tin cans containing antifriction bearings and inner packaging of varied cushioning value when the packages were subjected to rough handling during 3 weeks of cyclic exposure. The can packages in groups of 48 were packed by 2 methods in fiberboard inner boxex with outer boxes of wood.

No significant mechanical damage to the cans was caused by rough handling. All bearings were unaffected by the cyclic exposure and rough handling. Immediately after testing, however, the procoating on all cans contained a large number of water-filled blisters, most of which disappeared after a few days of exposure to 30 percent relative humidity. This blistered condition could be a principal factor in causing severe corrosion to the cans in the case of a longer period of exposure.

TR 53-38, Part 14 (Uncl)

January 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 14 - PERFORMANCE OF TIN CANS CONTAINING ANTIFRICTION BEARINGS AND COMMERCIAL MOTOR OILS (SERIES E-E)

INVESTIGATOR: A. A. Mohaupt

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture

ABSTRACT: To determine if commercial motor oils could be used for packaging antifriction bearings, tin cans containing separable-type ball bearings were filled or half filled with oil and sealed. Three motor oils, each of various viscosities, and an inhibited oil were used for these tests. The oils were applied at either 230° F. or room temperature. The cans were procoated with an olive-drab paint, and the packages were subjected to a 3-week cyclic exposure plus 60 days storage at 160°F. and 92 percent relative humidity.

Bearings packaged in an inhibited oil were in better condition after exposure than those packaged in motor oils. The different viscosities of the motor oils, filling or partially filling the tin cans, and the temperature at which the oil was applied to the bearings at the time of packaging had little or no effect on the condition of the bearings after exposure.

TR 53-38, Part 17 (Uncl)

October 1953

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 17 - THE PERFORMANCE OF TIN CANS CONTAINING TWO ANTIFRICTION BEARINGS PER CAN WITH VARIOUS INNER PACKAGING

INVESTIGATOR: R. K. Stern

CONTRACT: AF 18(600)-103

CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture

ABSTRACT: Tin cans, each containing two antifriction bearings, were subjected to a three week cyclic exposure plus 120 days storage at 160°F and 92 percent relative humidity.

Ball bearings with steel, fiber, or brass retainers were coated either with a synthetic grease, Specification MIL-G-3278 (Grease; Aircraft and Instruments, for High and Low Temperatures) or petroleum preservative, Specification AN-C-124 (Compound; Soft Film, Corrosion-Preventive). The bearings were then sealed in cans; or, after being coated, wrapped with each of 10 different intimate wrapping materials and sealed in cans; or, after being coated, sealed in cans which were filled with the petroleum preservative or synthetic grease.

Results indicate that the preservation of two bearings within a can with proper packaging materials is practical and compares favorably with results obtained where one bearing is preserved in a can with similar packaging materials.

The synthetic grease performed more effectively as a preservative than the petroleum preservative. Very good results were obtained from the cans filled with the preservative or grease. The most effective intimate wraps used with the synthetic grease were: cellulose acetate-kraft (plain or creped), waxed kraft, polyester film, cellulose acetate, and polyethylene. Wraps that produced the best results, when used with the petroleum preservative were: creped cellulose acetate-kraft, polyester film, and polyethylene.

TR 53-38, Part 18 (Uncl)-

January 1954

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 18 - THE EFFECT OF RELATIVE HUMIDITY AT TIME OF PACKAGING ON THE CONDITION OF BEARINGS AFTER A CYCLIC EXPOSURE (SERIES G-G)

INVESTIGATOR: R. A. Aubey  
CONTRACT: AF 18(600)-103  
CONTRACTOR: Forest Products Laboratory, United States Department  
of Agriculture  
ABSTRACT: Bearings were cleaned and packaged in metal cans at each  
of 6 humidity conditions ranging from 30 to 97 percent relative humidity.  
The intimate wraps, 1 hygroscopic (polyethylene to kraft) and the other  
nonhygroscopic (a polyester film), 3 preservative compounds, and 1  
grease were conditioned for 18 hours prior to packaging at the humidity  
in which they were to be used.

The packages were exposed to a 3-week cycle plus 60-days  
exposure at 160°F. and 92 percent relative humidity.

The condition of the bearings after exposure became  
increasingly worse as the humidity condition at time of packaging increased.  
When packaged at relative humidities of 50 percent and higher, bearings  
wrapped with the nonhygroscopic intimate wrap were in better condition  
than those with the hygroscopic wrap.

One of the petroleum-base preservative materials, when  
packaged with a polyester film as an intimate wrap, gave complete protection  
through all of the humidity conditions.

TR 53-38, Part 19, (Uncl)

July 1953

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 19 - PERFOR-  
MANCE OF TIN CANS CONTAINING ANTIFRICTION BEARINGS AND  
VARIOUS GREASES AND PRESERVATIVES.  
INVESTIGATOR: R. K. Stern  
CONTRACT: AF 18(600)-103  
CONTRACTOR: U. S. Forest Products Laboratory  
ABSTRACT: Tin cans containing antifriction bearings prepared with  
various amounts of grease or preservative were subjected to a 3-week cyclic  
exposure plus storage for 60 days at 160°F. and 92 percent relative  
humidity.

Two of the preservatives complied with Specification  
MIL-C-11796 Corrosion Preventive, Petrolatum, Hot Application and three  
with Specification AN-C-124, Compound; Soft Film, Corrosion Preventive;  
the two greases complied with Specification MIL-G-3278, Grease; Air-  
craft and Instruments (For Low and High Temperatures). Two proprietary  
compounds of the petrolatum type were also tested.

The bearings were either dip-coated with grease or  
preservative before they were inserted in the cans or they were inserted  
in cans filled or half-filled with grease or preservative. The preserva-  
tives were applied at 165°F. ( $\pm 5^\circ\text{F.}$ ) or at 225°F. ( $\pm 5^\circ\text{F.}$ ). The greases  
were applied at approximately 80°F.

Immersion of the bearings in greases or preservatives was more effective than dip-coating for preventing corrosion and staining. Cans, whether full or half-full, performed equally, except that more satisfactory results were obtained in the case of grease, Specification MIL-G-3278, when the cans were full. Equally satisfactory results were obtained with preservative, Specification MIL-C-11796, regardless of the temperature of application. However, where preservative, Specification AN-C-124 was used, better results were obtained with the 225°F temperature of application. All bearings preserved with the proprietary compound by any method were free of stain and corrosion.

TR 53-38, Part 20 (Uncl)

July 1953

SUBJECT: PACKAGING REQUIREMENTS FOR BEARINGS. PART 20 -  
ANTIFRICTION BEARING CAN PACKAGES SUBJECTED TO CYCLIC  
EXPOSURE AND 120 DAYS OF STORAGE

INVESTIGATOR: R. K. Stern

CONTRACT: AF 18(600)-103

CONTRACTOR: U. S. Forest Products Laboratory

ABSTRACT: Tin cans containing single antifriction bearings with various inner packaging were subjected to a 3-week cyclic exposure plus storage for 120 days at 160°F. and 92 percent relative humidity.

Separable-type ball bearings with steel, fiber, or brass retainers were coated with either a synthetic grease or a petroleum-base preservative, wrapped with 1 of 13 different intimate wraps, and sealed in cans. In addition, coated bearings of each type were packaged without intimate wraps and sealed in cans, and others were sealed in cans that were filled with preservative or grease. The can exteriors were spray-coated to varied thicknesses with one of two kinds of paint.

All bearings packaged without intimate wraps and sealed in cans filled with grease or preservative were free from corrosion and stain after testing. Good results were also obtained when intimate wraps of nylon, cellulose acetate-kraft, or polyethylene-kraft were used with synthetic grease, Specification MIL-G-3278, Grease; Aircraft and Instruments (For Low and High Temperatures). In general, erratic results were obtained when the preservative conforming to Specification AN-C-124, Compound; Soft Film, Corrosion Preventive, was used.

The best over-all results were obtained with the pro-coating, when paint that conformed to Specification MIL-C-10506, Coatings, Exterior, for Tinned Food Cans, was sprayed on the cans to an average dry film thickness of 1.3 mils.

TR 53-132 (Uncl)

January 1954

SUBJECT: THE DESIGN OF RECLAIMED LATEX FOAM PACKAGE CUSHIONING.  
THE THIRD OF A SERIES OF REPORTS ON PACKAGE CUSHIONING  
INVESTIGATOR: Roger B. Orensteen, 1st Lt, USAF  
CONTRACT: RDO No. 618-11  
ABSTRACT: A method of designing reclaimed latex foam cushioning from static data is presented. Design curves are provided for selecting density, thickness, and cushion area for economical cushion design. Recommended procedures are discussed for designing to optimum density, stress, and ratio/stress energy in a given situation.

TR 53-133, Part 1 (Uncl)

June 1954

SUBJECT: INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES  
INVESTIGATOR: Jules Pinsky, A. R. Nielsen and James H. Parlman  
CONTRACT: AF 33(616)-112  
CONTRACTOR: Plax Corporation  
ABSTRACT: The shelf life of 80 especially selected liquids and solids in blown Plax 4 ounce polyethylene bottles was investigated at temperatures from 32-165°F. Permeability factors for these materials are presented as well as packageability suitability. The suitability of packaging these 80 materials in blown polyethylene bottles of capacities from 32 ounces to 13 gallons is tabulated. Physical effects on the polyethylene and chemical changes in the materials are described. The effect of liquid and vapor phases, as well as the molecular weight of the chemicals, on permeability is discussed.

TR 53-216 (Uncl)

January 1954

SUBJECT: EVALUATION OF CONTAINER-GRADE PAPER-OVERLAID VENEER  
INVESTIGATOR: E. H. Clarke  
CONTRACT: AF 33(038)51-4065  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: By conducting over 1,500 tests on specimens of paper-overlaid veneer and container-grade plywood along with tests of 165 containers, information was obtained that indicates a correlation exists between some of the mechanical properties of paper-overlaid veneer materials and the expected rough-handling performance of fully cleated domestic boxes using such material for panels. The results also indicate that all of the tested 19 different paper and veneer combinations, each varying from the other by differences in core, overlays, or manufacturing process, are suitable alternates for 3/20-inch (Groups I and II) container-grade plywood in panel boxes. Some combinations appeared to be suitable as alternates for thicker plywood.

January 1954

SUBJECT: CALCULATING CUSHION THICKNESS BY ANALYSIS OF STRESS-STRAIN CURVES  
INVESTIGATOR: R. E. Jones and D. L. Hunzicker  
CONTRACT: AF 33(038) 51-4065  
CONTRACTOR: Forest Products Laboratory, United States Department of Agriculture  
ABSTRACT: A method for designing package cushioning to protect an article against shock is developed from physical and mathematical concepts. Design curves for calculating cushion thickness are included for many currently available cushioning materials. Methods for selecting the proper cushion are discussed.

PETROLEUM PRODUCTS, ANTISEIZE COMPOUNDS

February 1954

SUBJECT: DEVELOPMENT OF TEST METHODS FOR ANTISEIZE COMPOUNDS  
INVESTIGATOR: John W. Cunningham  
CONTRACT: AF 33(038)-22805  
CONTRACTOR: Southwest Research Institute  
ABSTRACT: Initial development of a tapered pin seizure test method and testing device to evaluate antiseize compounds (WADC TR 52-102, May 1952) indicated the need for modification of both method and device in order to increase sensitivity, determine the effect of certain variables on the effectiveness of antiseize compounds, simplify the comparison of individual test results, and determine the validity of the tapered pin test method as a means of predicting antiseize compound performance under actual field conditions. To meet these requirements, the tapered pin seizure tester was modified to provide a progressively increasing contact pressure. Tests were conducted to determine the relationship to the effectiveness of antiseize compounds of varied time and temperature exposure, different specimen materials, various specimen surface finishes, specimen assembly design modifications, and vibration at varying frequencies and amplitudes. Visually plotted averages of individual test results were transposed to rectangular coordinates. Antiseize compounds applied to both tapered pin and threaded specimen assemblies of identical materials were tested under identical time and temperature conditions to determine the extent to which the test results were correlative.

In general, the modified and improved tapered pin seizure test method provides more extensive information of greater validity regarding the effectiveness of antiseize compounds. It is believed, however, that the validity and reliability of the data obtained could be greatly increased by further development to facilitate torque measurement, to

permit the direct recording of test data on rectangular coordinates, to assure more accurate transmission of thrust load, and to provide a continuous increase in contact pressure.

PETROLEUM PRODUCTS FUELS

TR 52-35, Supplement 1 (Uncl)

January 1954

SUBJECT: RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS  
INVESTIGATOR: G. W. Jones, M. G. Zabetakis, G. S. Scott and A. L. Furno  
CONTRACT: AF 18(600)-151  
CONTRACTOR: United States Department of the Interior  
ABSTRACT: The results of limit of flammability, limit of ignitibility, and ignition temperature tests conducted on aircraft fuel vapor-air mixtures, with various quantities of added nitrogen and carbon dioxide, by the U. S. Bureau of Mines Gas Explosions Branch between 1 February 1952 and 1 February 1953 are presented. Two aviation gasolines grades 100/130 and 115/145, and three jet fuels grades JP-1, JP-3 and JP-4 were used in the investigations.

TR 53-63 (Uncl)

November 1953

SUBJECT: STORAGE STABILITY OF JET TURBINE FUELS  
INVESTIGATOR: C. A. Cole and A. C. Nixon  
CONTRACT: AF 18(600)-37  
CONTRACTOR: Shell Development Company  
ABSTRACT: This investigation was initiated to ascertain the variables influencing the storage stability of jet fuels and to determine the simplest and most practical methods of controlling these variables for a resultant increase in the permissible storage life of the fuels. The work is being done both under laboratory aging and desert field storage conditions. An early program under Navy sponsorship (Contract No. (NOa(S) 9970)) surveyed and developed the various methods of determining and measuring stability of jet fuels. With development of the 500°F steam jet gum evaporation unit and the use of the accelerated aging procedure, it was decided that reasonable predictions of stability in terms of field storage could be made if correlations between field and laboratory aging conditions were established. A subsequent Air Force Contract (No. AF 33(038)-7277) led to the limited determination of these relationships as well as the study of various factors influencing stability, such as fuel type, contamination, inhibition, treatment, storage temperature and minor components. During the present contract, work on all of these phases of study was continued and extended to include the effects of gum on fuel filterability, dielectric constant and blending compatibility.

The results of the present study may be summarized as follows: Both soluble and metallic copper have an adverse effect on stability. Sea water in the presence of iron appears to promote insoluble

gum formation; however, water, generally, reduces soluble gum formation. High boiling materials contribute more to the instability of a blend than do low boiling materials. The average thermally cracked fuel is more unstable than the average catalytically cracked fuel. Some sulfur and nitrogen compounds catalyze gum formation.

A large number of compounds have been tested as potential jet fuel inhibitors. None tested, including the specification inhibitors, have shown consistent stabilizing action.

The freezing point of a fuel can change with aging of the fuel, but no correlation exists between the quantity of gum formed and the freezing point change. No simple relationship was observed between stability and unsaturation, conjugated diolefins, nitrogen, sulfur or peroxide content.

Correlations between accelerated and desert aging for all fuels tested indicate that 5 hours' aging (at 100°C and 100 psig O<sub>2</sub>) is about equivalent to two years of desert drum storage. Fuels correlated on the basis of type show wide variations from this relationship, however.

A method for determining the constant flow filterability characteristics of jet fuels at any practical temperature has been developed which required only 100 ml of fuel. Although only scant data are available as yet, the method appears to give good correlation with large scale results.

On the basis of present data, soluble gum does not appear to affect filterability characteristics. Water, if not present above the saturation point of the fuel, does not affect filterability at temperatures as low as -30°C (no data are available with excess water). Insoluble gum is important in determining the filter clogging tendencies of a fuel, but no relationship exists between the quantity of insolubles present and the tendency of the fuel to clog a filter.

It was found that in some cases quite small quantities of insoluble gum could markedly influence the filter clogging effect of a fuel. This effect was strongly accentuated by lowering the temperature.

Moderate quantities of gum do not affect the dielectric constant of a fuel significantly.

Compatibility of various components of a blend is not affected in most cases by a moderate amount of aging before blending.

This investigation is continuing with emphasis on methods of improving stability by inhibition and treatment utilizing practical jet fuel components and blends and model compounds.

PETROLEUM PRODUCTS, HYDRAULIC FLUIDS

TR 6685, Suppl 2 (Uncl)

September 1953

SUBJECT: DEVELOPMENT OF HIGH-TEMPERATURE AIRCRAFT HYDRAULIC FLUIDS  
INVESTIGATOR: Neil W. Furby  
CONTRACT: AF 33(038)9831  
CONTRACTOR: California Research Corporation  
ABSTRACT: Progress for the year 1952 in the development of a high temperature aircraft hydraulic fluid for operation from  $-65^{\circ}\text{F}$  to  $400^{\circ}\text{F}$  is described. Compounds synthesized for investigation as base materials included silicates, trisiloxanes, silicon-phosphorus compounds, phosphonates, and miscellaneous compounds. Diethyl and methylphenyl silicones as well as polymethacrylates were investigated as V.I improvers for silicate base fluids. Additives were evaluated for effects on high temperature stability, hydrolytic stability, and antiwear properties. Four-ball wear tests and 3000 psi Vickers and New York Airbrake piston pump tests were conducted on several experimental fluids to determine lubricating properties.

TR 53-462 (Uncl)

April 1954

SUBJECT: CHLORINE-AND FLUORINE-CONTAINING COMPOUNDS FOR NONFLAMMABLE MATERIALS  
INVESTIGATOR: O. R. Pierce and E. T. McBee  
CONTRACT: AF 18(600)-128  
CONTRACTOR: Purdue University  
ABSTRACT: Flash point, fire point and autogenous ignition temperature were determined for mixtures of 25 bromine compounds with hydraulic fluid MIL-0-5606. None of the additives raised the autogenous ignition temperature of the hydraulic fluid appreciably. Flash point and fire point were increased by the addition of bromo-form, 1,1,2,2,-tetrabromoethane, 1,3-dibromopropane and carbon tetrabromide.

A series of alkylphosphonosilanes and alkylphosphonoethoxysilanes was prepared from alkylchlorosilanes and appropriate phosphorus compounds. The entire group exhibited good viscosity and resistance to burning but relatively poor hydrolytic stability. The compound  $[(n\text{-BuO})_2\text{P}^{\text{O}}]_2\text{SiEt}_2$  (code No PS-52) was found to possess superior viscosity properties and good non-flammability.

Fluorine-containing polyethers were obtained from  $\text{F}_3\text{CCH}(\text{O})\text{CH}_2$  and sodium ethoxide. A fraction of the polymer, presumably  $\text{H}(\text{OCH}(\text{CF}_3)\text{CH}_2)_2\text{OEt}$ , was unsatisfactory in all respects as hydraulic fluid.

The ester  $(\text{CF}_3\text{CF}_2\text{CF}_2\text{CH}_2\text{O})_4\text{Si}$  was prepared from silicon tetrachloride and heptafluorobutanol. It has a high flash point, fire point and autogenous ignition temperature.

Fluorine-containing esters and acids were prepared as intermediates from polyfluoroaldehydes by use of the Reformatsky reaction with  $\alpha$ -bromo esters. The ionization constants of the acids were determined and interpreted in terms of electronic effects. Both saturated and unsaturated esters were converted to alcohols through reduction.

Fluorinated phosphonate esters  $\text{RPO}(\text{OCH}_2\text{CF}_3)_2$  were obtained from bis-trifluoroethyl chlorophosphate by action of Grignard reagents or from tris-trifluoroethyl phosphite and alkyl lithium compounds and air oxidation of the resultant alkyl phosphonites. The compounds were found to have poor hydrolytic stability.

Perfluoroalkyllithium compounds were prepared by lithium-halogen interchange at low temperature and were utilized in addition reactions with carbonyl compounds.

The reaction between ethyl orthosilicate and heptafluoropentylmagnesium bromide was further investigated. The compounds  $(\text{C}_3\text{F}_7\text{CH}_2\text{CH}_2)_4\text{Si}$  and  $(\text{C}_3\text{F}_7\text{CH}_2\text{CH}_2)_2\text{Si}(\text{OEt})_2$  were separated from the products and purified. The latter was polymerized by heating with hydrochloric acid. A distilled fraction of the polymer showed good resistance to burning (high AIT) but very poor viscosity properties.

Fluorine-containing alcohols were prepared as intermediates from fluoroalkylmagnesium halides by reaction with oxygen and paraformaldehyde. By direct oxidation of these primary alcohols the corresponding acids were produced in good yield. The degradation of their silver salts with bromine furnished fluoroalkyl bromides. The esters of the fluorinated acids were converted to secondary alcohols by reaction with primary alkylmagnesium halides and isopropylmagnesium bromide.

PETROLEUM PRODUCTS, LUBRICANTS

TR 53-88 (Uncl)

October 1953

SUBJECT: SYNTHETIC LUBRICANTS  
INVESTIGATOR: W. E. McTurk  
CONTRACT: AF 33(038)-14593  
CONTRACTOR: Standard Oil Development Company  
ABSTRACT: This report is a compilation of all the information obtained under Contract No. AF 33(038)-14593, concerned with means of increasing the availability of synthetic lubricants for use at low and high temperatures.

The diesters of straight-chain dibasic acids lead the field of esters suitable as lubricants for use at both low and high temperatures, because of their desirable combinations of properties and potentially good availability. Adipic, azelaic, and sebacic acids are the most readily available dibasic acids suitable for ester lubricant production, while the petroleum derived Oxo alcohols appear to be the most available alcohols for this application. In addition, however, certain diesters of polypropylene glycols appear equivalent to dibasic acid esters in all the characteristics studied so far, and this type of ester therefore represents a promising source of synthetic oil. Mono-esters may be satisfactory lubricants where high temperatures are not encountered. The presence of small quantities of impurities are believed to cause variations in the oxidation and thermal stability of esters. The SAE E.P. Lubricants Tester is capable of measuring the load-carrying ability of synthetic lubricants, but further work is required to develop a completely reliable test for predicting full-scale gear performance.

TR 53-293 Part 1 (Uncl)

October 1953

SUBJECT: HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC-BASE OILS  
PART 1 - TESTING METHODS AND PRELIMINARY RESULTS  
INVESTIGATOR: James W. Cole, Jr., Arthur F. Benton, Alfred Burger  
and Thomas I. Crowell  
CONTRACT: AF 33(038)-22947  
CONTRACTOR: University of Virginia  
ABSTRACT: PHASE I. Literature Survey. The literature on oxidation and corrosion inhibitors for the various oils was completely surveyed for the period 1916 to date. There seem to be no pertinent references earlier than 1916. Each of the approximately 2500 references to research reports, patents, reviews, etc. was recorded and coded on a McBee Key-sort punch card. Three punch card indices were devised covering 105 entries, each being designated by a four letter code word. A novel card needling box was designed and constructed to facilitate the use of the punch card file. A detailed report on the literature will be the subject of a separate memorandum report.

PHASE II. Evaluation of Additives and Mechanism Studies. With a specially constructed aluminum block thermostat, 60 likely-looking compounds of various types were tested as oxidation-corrosion inhibitors for di-(2-ethylhexyl) sebacate (Rohm and Haas, Plexol 201). Substances of the phenothiazine-type were the most effective at 400°F. These types were exhaustively studied and the effects of concentration, small changes of temperatures, metals, and substituents shown. Probably the most effective compounds were N-methyl- and N-ethyl-phenothiazine. At 400°F a concentration of about 1.6% is needed to hold the oxidation induction period for 72 hours in the presence of copper.

Systematic studies were started on the mechanisms of pyrolysis and oxidation of diesters, the chemistry of phenothiazine and the mechanism of its action, the rate of depletion of additives, and the role of metals in the oxidation phenomena. Each of these is of a complex nature and only tentative conclusions were reached.

PHASE III. Research and Development of New Materials. Efforts were directed toward improving syntheses of some of the substances tested in Phase II and preparing new compounds together with the study of properties of the more interesting compounds. Work was under the following headings: Purification of phenothiazine; preparation of p,p'-dihydroxydiphenylamine, p-benzyloxydiphenylamine, 3-benzyloxyphenothiazine, p-t-butoxydiphenylamine, 10-methylphenothiazine, 3-formyl-10-methylphenothiazine. 3-formyl-10-methylphenothiazine oxime, 3-(10-methylphenothiazinyl)methanol,  $\beta$ -[3-10-methylphenothiazinyl] acrylic acid, 1-nitro-2-[3-(10-methylphenothiazinyl)]ethene, phenyl-beta-naphthylamine and 7-benzo(c)phenothiazine; nitration of 7-benzo(c) phenothiazine; preparation of N-acetyl-7-benzo(c) phenothiazine; nitration of N-acetyl-7-benzo(c) phenothiazine; Friedel-Crafts acetylation of N-acetyl-7-benzo(c) phenothiazine; preparation of x-acetyl-7-benzo(c)phenothiazine oxime; attempted rearrangement of the oxime of x-acetyl-7-benzo(c)phenothiazine; preparation of N-methyl-7-benzo(c)phenothiazine, N-methyl-7-benzo(c)phenothiazine-S-oxide, 7-benzo(c)phenothiazine-S-oxide and x-carboxy-7-benzo(c)phenothiazine.

TR 53-293, Part 2 (Uncl)

December 1953

SUBJECT: HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS.  
PART 2 - EVALUATION OF ADDITIVES, SYNTHESSES OF NEW COMPOUNDS, AND MECHANISM STUDIES

INVESTIGATOR: James W. Cole, Jr., Gordon P. Brown, Alfred Burger and Thomas I. Crowell

CONTRACT: AF 33(038)-22947

CONTRACTOR: University of Virginia

ABSTRACT: A total of approximately 200 compounds has been evaluated as oxidation-corrosion inhibitors in various ways in a total of 13 synthetic base oils. The base oils used include diesters, phosphonates, silicates and silicones.

Various methods of comparing effectiveness of additives are discussed. There is as yet no one satisfactory method for rating a particular compound in a specific oil.

Phenothiazine and some of its derivatives remain the best inhibitors, as a class, for the diesters at 204.4°C. Preliminary results suggest that phenothiazone-3 may be the best of this type. Two N-alkyl-alpha-naphthylamine derivatives, N-methyl- and N-ethyl-, also show good inhibitor properties in di (2-ethylhexyl) sebacate.

Many rubber antioxidants, particularly those containing sulfur, showed good antioxidant properties in the diester. However, these attacked copper in excess of that allowed in the specifications.

In mixed C<sub>6</sub>-C<sub>8</sub> silicate and tetra (2-ethylhexyl) silicate, N-phenyl-alpha-naphthylamine continues to be one of the most effective oxidation inhibitors. Preliminary tests indicate that N-ethyl-alpha-naphthylamine, some p-aminophenols and some p-phenylenediamines may be of equal activity.

DC Silicone 550 apparently needs no oxidation-corrosion inhibitors to meet the specifications at 204.4°C since it has an induction period in excess of 75 hours. Indeed, the change in measured properties of DC Silicone 550 is generally greater in the presence of those additives tested than it is in the absence of additives. This increase in apparent oxidation in the presence of additives might be assumed to be due to changes in the additives.

Mechanism studies have been conducted on the action of additives and the media under conditions of oxidation, pyrolysis and metal catalysis. The products of pyrolysis of di (2-ethylhexyl) sebacate in the range 260°-305°C over the first 10% of the reaction have been identified as 2-ethylhexene-1 and the mono-ester, 2-ethylhexyl hydrogen sebacate.

Studies have been made on the rate of disappearance of phenothiazine in di (2-ethylhexyl) sebacate in the presence and absence of copper, either as the metal or as a salt. The rate of disappearance of phenothiazine is slowed in the presence of copper but the length of induction period is shortened. An attempt is made to explain this phenomenon on the basis of accepted reaction mechanisms. It is also shown that the rate of disappearance of phenothiazine closely resembles first-order kinetics.

TR 53-293, Part 3 (Uncl)

December 1953

SUBJECT: HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. PART 3 -  
THE THERMAL DECOMPOSITION OF DI-2-ETHYLHEXYL)SEBACATE.

INVESTIGATOR: Earl E. Sommers and Thomas I. Crowell  
CONTRACT: AF 33 (038)-22947  
CONTRACTOR: University of Virginia  
ABSTRACT: A brief survey of the literature is given describing the postulated mechanisms by which esters of the type in question decompose. The determination of the rate of thermal decomposition of di-(2-ethylhexyl) sebacate in the liquid phase in the temperature range of from 260° to 305° C is described. Conclusions to be drawn are that the decomposition proceeds only at a very slow rate at 204° C, yet it does occur; and that it is a simple reaction, i.e. not of the free radical type, nor is it affected by acids or a copper surface.

From the literature it is concluded that certain ester types which might be expected to be stable at these temperatures should be prepared and subjected to conditions analogous to, or more drastic than those described herein.

TR 53-393, Part 4 (Uncl)

December 1953

SUBJECT: HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS.  
PART 4 - STUDIES IN THE COPPER-PHENOTHIAZINE Di-(2 ETHYL-  
HEXYL) SEBACATE-SYSTEM IN THE NEIGHBORHOOD OF 200° C (400° F).

INVESTIGATOR: James W. Cole, Jr. and Lewis G. Cochran  
CONTRACT: AF 33(038)-22947  
CONTRACTOR: University of Virginia  
ABSTRACT: This is a detailed study at several temperatures in the neighborhood of 200° C of anomalous behavior encountered in the oxidation studies of diester lubricants in the presence of phenothiazine and copper and was designed to shed more light on the mechanisms of inhibitor action and metal catalysis. The behavior of phenothiazine in di (2-ethylhexyl) sebacate under controlled oxidation conditions was followed by noting the change in the characteristic ultraviolet absorption spectra and by chemical methods. The rates of decrease of absorption of phenothiazine at its characteristic maximum, 2520 Å, indicated that phenothiazine disappeared less rapidly in presence of copper than when this substance was absent, even though the overall oxidation as measured by rate of oxygen absorption was more rapid. The effect of copper salts of the dibasic acids, sebacic and oxalic, was of the same order, but the detailed effect of concentration of the dissolved salts was complex. The results were interpreted in terms of possible reaction mechanisms. It appears that phenothiazine is rapidly converted to an intermediate oxidation state of finite life through energy-rich chains formed in the medium. The slower rate of disappearance in the presence of copper apparently arises from the action of the metal converting the energy-rich chain carriers relatively faster into free-radical chains and thereby reducing the concentration of the energy-rich units which act to convert phenothiazine to the intermediate oxidation state. The net effect in the presence of copper is that phenothiazine has less

opportunity to become activated while at the same time the active form is dissipated more rapidly and inactive oxidation products form at a faster rate. A rate equation is derived for the disappearance of phenothiazine and the mechanisms proposed are related to the possibility of predicting antioxidants for higher temperatures.

TR 53-303 (Uncl)

November 1953

SUBJECT: LOW-TEMPERATURE, HEAT AND OXIDATION STABLE MATERIALS  
AS POSSIBLE LUBRICANTS AND ELASTOMERS  
INVESTIGATOR: Murray Hauptschein and Charles S. Stokes  
CONTRACT: AF 33(038)-10844  
CONTRACTOR: The Research Institute of Temple University  
ABSTRACT: As part of the program of correlating properties with structural features of various types of fluorinated ester-type compounds, a good deal of research was carried out on devising methods for the syntheses of di-, tri-, tetra- and poly- esters.

There have been synthesized sizeable quantities of seven polyethylene glycol di-n-perfluorobutyrate and a polypropylene glycol di-n-perfluorobutyrate. In addition the nonfluorinated analogs of the polyethylene glycol di-n-perfluorobutyrate have been prepared for purposes of comparison.

The synthesis of 1,5-di-(2'-cyanoethoxy) pentane and the attempted conversion of this compound to 1,5-di-(2'carboxyethoxy) pentane is given.

Two unsuccessful attempts to prepare 1-iodo-1,1,2,2-tetrahydroperfluoropentane by the reaction of 1-iodoheptafluoropropane with ethylene are described.

The preparation of the triester, 2-2'-hydroxyethoxymethyl)-2,4-dimethyl pentanediol-1,5 triperfluorobutyrate and the attempted preparation of 2-nitro-2-hydroxymethyl propanediol-1,3 triperfluorobutyrate are reported.

The synthesis of two half-ester acids, namely, mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate and mono-2-ethyl-n-hexyl perfluoroglutarate made possible the preparation, in good yields, of four pure tetraesters. These compounds are: (1) pentamethylene dithiol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (2) pentamethylene glycol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (3) trimethylene glycol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (4) trimethylene glycol bis (mono-2-ethyl-n-hexyl perfluoroglutarate).

Due to the very great difficulty in the preparation of pure mono-esters of glycols of the types  $C_3F_7COO(CH_2)_nOH$  and  $C_3F_7COOCH_2CH_2OCH_2CH_2OH$ , as well as in the preparation of pure half ester-acids such as  $C_3F_7CH_2OOC(CH_2)_4COOH$ , only impure tetraesters could be obtained on reacting these intermediates with various dicarboxylic acids and glycols, respectively.

The completely branched tetra ester, pentaerythritol monocaproate triperfluorobutyrate has been synthesized.

The preparation and properties of the polyester, poly-(diethylene glycol perfluoroglutarate) are reported.

Correlations on the viscosity and surface tension characteristics of the various series of ester-type materials have been made.

TR 53-337 (Uncl)

November 1953

SUBJECT: POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS  
INVESTIGATOR: Charles F. Raley, Jr.  
CONTRACT: AF 33(616)-276  
CONTRACTOR: Southwest Research Institute  
ABSTRACT: A literature survey covering the field of polynuclear aromatic compounds, with two or three nuclear rings, was made. All materials liquid at 35°C. and stable up to 400°F. were entered on file cards. From the data thus gathered, a list of seventeen compounds, considered to be representative of the common chemical structures, was compiled. These compounds were synthesized and evaluated as to thermal stability, viscosity at 100°F., 210°F., density, and boiling point. On the basis of this evaluation, the field of aryl phosphates was considered to have the most likely chance of providing compounds with the desired properties. Further research in this field has produced three liquid compounds with exceptional thermal stabilities, i.e., above 700°F.; di-p-tolyl 1-naphthyl phosphate, di-m-tolyl 1-naphthyl phosphate, and tri-o-chlorophenyl phosphate. In the case of the first two compounds, all other screening tests, such as flash and fire point, autogenous ignition temperature and hydrolytic stability, gave excellent results. Other compounds which will be synthesized offer a reasonable chance of possessing even better properties.

TR 53-353 (Uncl)

May 1954

SUBJECT: SURVEY OF THE LITERATURE ON ANTIOXIDANTS AND ANTI-CORROSION ADDITIVES FOR LUBRICANTS AT ELEVATED TEMPERATURES  
INVESTIGATOR: James W. Cole, Jr., Alfred Burger and Arthur F. Benton  
CONTRACT: AF 33(038)-22947  
CONTRACTOR: University of Virginia

ABSTRACT: The literature was surveyed on oxidation and corrosion inhibitors in various media at elevated temperatures. Particular attention was given to phenothiazine types of additives and to lubricants of the synthetic-base type. Media, such as rubber, were included where the effects noted might have analogies in synthetic base lubricants. The literature revealed very few antioxidant or anticorrosion combinations for synthetic base lubricants at extreme temperatures. The survey covered the period 1916 to June 1952. There were very few significant literature references prior to 1916. The survey can be readily brought up-to-date when the 1952 subject index of Chemical Abstracts becomes available. Approximately 2500 abstracts of research reports, patents, reviews, articles, etc. were recorded on McBee Keysort Punch Cards of special design. Three punch card indices were devised covering approximately 100 entries, each entry being designated by a four-letter code word. A novel card-needing box was designed and constructed to facilitate use of the card file.

TR 54-44 (Uncl)

April 1954

SUBJECT: INVESTIGATION OF "BENTONE-34" AS A GREASE THICKENER  
INVESTIGATOR: Herbert Schwenker  
CONTRACT: RDO No. 613-11(E-A)  
ABSTRACT: The project was initiated to develop greases from domestic, non-critical materials that will ensure the Air Force a supply of greases whose source will not be limited or cut off because of war or other emergencies.

"Bentone 34" was used as a thickening agent with various synthetic and petroleum oils in preparing greases. The greases were tested for their physical and chemical properties as possible aircraft lubricants. The test results indicated that it may be possible to formulate aircraft lubricating greases using "Bentone 34" as a thickener which will be equal in performance to the soap-type greases now in use.

#### PLASTICS, STRUCTURAL

TR 52-5, Supplement 2 (Uncl)

March 1954

SUBJECT: HIGH STRENGTH EPON LAMINATES  
INVESTIGATOR: F. C. Hopper  
CONTRACT: AF 33(038)-19587  
CONTRACTOR: Shell Development Company  
ABSTRACT: Mixtures of one part EPON 1001 with 2 parts Plyophen 5023, cured with dicyandiamide and using 181-Volan A glass fabric, yielded laminates with good strength at room temperature and at elevated temperature after short-time exposure. Resistance to aging at 500°F was poor, but was better than that of Plyophen 5023 alone. Strengths typical of good samples follow:

Test Temp.,	Exposure,	Flexural,
<u>deg. F.</u>	<u>hrs.</u>	<u>ult., psi-</u>
Room	Indef.	70,000
300	1/2	55,000
500	1/2	45,000
500	200	7,000

A laminate so made passed the mechanical strength requirements of Military Specification MIL-R-7575A, but failed to meet the requirements of weight and thickness charges on immersion in water and various chemicals.

Fabric impregnated with mixtures of the two resins exhibited poor storage stability. Laminates made with fabric stored 35 days at room temperature were high in resin and had less than one-half of their initial flexural strength at 500°F after 1/2 hour at 500°F, although resistance to aging at 500°F was not impaired.

EPON 1004 could be used successfully in place of EPON 1001 and Lebec 102,210 was equal in performance to Plyophen 5023.

EPON X-12100 (P-2592) yielded laminates which were less strong than the foregoing on short-time exposure, but were more resistant to high temperature aging. After 200 hours at 500°F, the strength of one laminate at 500°F was 12,000 psi. This was increased to about 15,000 psi in other laminates by the addition of a small amount of copper 8-quinolinate as stabilizer. The addition of 10 to 20 PHR<sup>a</sup>) of Plyophen 5023 appeared to effect even better stability, yielding laminates having flexural strengths of 16,700 and 19,600 psi at 500°F after aging.

Laminates containing EPON X-003.12.0, cured with dicyandiamide, had the highest strengths so far obtained with an EPON resin at 300°F, (64,600 psi), but failed badly at 500°F.

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a) PHR = parts per hundred of resin and is equivalent to per cent based on the resin.

A new curing agent, know as "CL", greatly improved the elevated temperature strength of EPON 828. It could not be used with the more heat resistant solid EPON resins, such as EPON X-12100, which must be applied from solution, because it cured the resin during drying of the fabric to remove the solvent.

With the possible exception of EPON 828 cured with CL, EPON 1001 containing dicyandiamide continued to yield laminates of higher

room temperature strength than any other EPON resin. Wright Air Development Center TR No. 52-5; Supplement No. 1 describes this type of laminate with which flexural strengths of 76,900 psi and compressive strengths up to 70,200 psi at room temperature have been obtained.

TR 52-183, Supplement 1 (Uncl)

October 1953

SUBJECT: ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, "PLASTICS FOR AIRCRAFT"  
INVESTIGATOR: Donald G. Coleman  
CONTRACT: AF 33(038)-51-4326E  
CONTRACTOR: U. S. Department of Agriculture  
ABSTRACT: Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1953 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Four technical reports issued during the fiscal year are abstracted.

TR 53-56 (Uncl)

November 1953

SUBJECT: DEVELOPMENT OF FURANE RESIN GLASS FABRIC BASE PLASTIC LAMINATES  
INVESTIGATOR: John Delmonte, John Knudsen and Leland Smith  
CONTRACT: AF 33(600)-17708  
CONTRACTOR: Furane Plastics Incorporated  
ABSTRACT: Furane resins, prepared from abundant non-critical materials were used to fabricate glass fabric base plastic laminates for testing against the requirements of MIL-R-7575 Specification. In this report the influence of various catalysts upon the resin are shown. In the preparation of 1/8th inch thick laminates, furane resins activated with acid catalysts required post curing to achieve good physical properties. However, one proprietary catalyst, Reagent CM, yielded laminates with good physical properties without the necessity of post cure. No. 181 glass cloth with Garan finish was used exclusively. Differences are brought out between a stiff and soft finish.

To attain optimum results, laminating techniques were carefully evaluated upon small (6 in x 8 in) laminates including: precuring (optimum was 90 minutes at room temperature); storage or closed assembly (permissible up to 24 hours); contact time at laminating temperature (30 to 60 seconds was desirable); and breathing (generally within 30 to 60 seconds). The techniques required coordination to achieve best properties.

Low molecular weight and high molecular weight furane resins were examined, and Resin XL was developed in an attempt to meet MIL-R-7575 Specification requirements. Modifications with phenolic resins also showed promise. Flexural strengths of 60 to 65,000 p.s.i. and edgewise compressive strengths of 35 to 40,000 p.s.i. appeared readily obtainable on small laminates.

Laminates of large area (2 ft x 2 ft) gave lower and less consistent values for strength properties than the smaller laminates. The area in the center portion of the large laminates showed lower test values than those specimens nearest the edges.

TR 53-192, Part 2 (Uncl)

August 1953

SUBJECT: MECHANISM OF RAIN EROSION. PART 2 - A CRITICAL REVIEW OF EROSION BY WATER DROP IMPACT  
INVESTIGATOR: Olive G. Engel  
CONTRACT: Delivery Order No. 33(616)-53-9  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: A critical review of the literature on the subject of erosion by water drop impact which includes some recent work done in this laboratory has been made. The types of experimental apparatus generally used by the investigators, and the factors which they found determine the extent of the erosion damage, are briefly discussed. Results of microscope and X-ray studies are presented. Theoretical estimates of the impact pressure, the results of piezoelectric pressure measurements, and theories which have been advanced as to the mechanism of the erosion process are reviewed. Questions which are still unanswered, or in regard to which further research should be done, are pointed out.

The views and conclusions of the investigators are in many cases presented in their own words. A large number of the quotations are translations.

TR 53-192, Part 3 (Uncl)

December 1953

SUBJECT: MECHANISM OF RAIN EROSION. PART 3 - MECHANISM STUDIES ON PLASTICS AND METALS  
INVESTIGATOR: Olive G. Engel  
CONTRACT: AF 33(616)53-9  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: Rain erosion damage on brittle materials of low tensile strength has been reproduced in a parallel study of the damage caused by the impact of steel spheres and of deforming lead pellets on methyl methacrylate plastic. Analysis of the damage in the parallel study involving steel spheres and lead pellets has essentially clarified the mechanism of rain erosion on brittle materials of low tensile strength.

Investigation made with metals include preliminary studies of the effect of polish, of general hardening, and of surface hardening. Although clues have been found which seem to explain how the erosion progresses on metals after it has been initiated, the precise mode of formation of the first pit nucleus has not been ascertained. Three possible modes of formation are discussed.

January 1954

SUBJECT: MECHANISM OF RAIN EROSION. PART 4 - CAVITATION AS A RESULT OF WATERDROP COLLISIONS WITH SOLID SURFACES

INVESTIGATOR: Olive G. Engel

CONTRACT: AF 33(616)53-9

CONTRACTOR: National Bureau of Standards

ABSTRACT: A search was made to determine whether or not cavitation takes place in a waterdrop when impact occurs between the drop and a solid surface. Five arrangements of the lights and camera were tried in an effort to obtain maximum resolution in high speed moving pictures of any bubbles which may form. Some positive evidence was obtained showing that cavitation does occur. Further tests with carbon dioxide-saturated water and with argon-saturated water, which are now planned, should either reinforce or nullify this evidence. The wash configurations which result from impact of a waterdrop against surfaces having different degrees of smoothness, and against materials having different degrees of resilience, were observed with high speed moving pictures.

January 1954

SUBJECT: FLEXURAL TESTS OF STRUCTURAL PLASTICS AT ELEVATED TEMPERATURES

INVESTIGATOR: John E. Wier and Dorothy C. Pons

CONTRACT: AF 33(038)-51-4358

CONTRACTOR: National Bureau of Standards

ABSTRACT: Flexural strength and modulus of elasticity at elevated temperatures were determined on 1/8 inch thick glass-fabric laminates bonded with three types of commercially-available heat resistant resins. The resins were: 1) a silicone, DC 2104; 2) a polyester, PDL-7-669; and 3) a phenolic, CTL-91LD. The laminates were made of glass-fabric, Fiber-glas 181 with finishes 112 (heat cleaned), Garan, and 114. These finishes were used with the silicone, polyester, and phenolic resins, respectively.

The laminates were exposed to temperatures of 300, 400, 500, 600, and 700°F for periods ranging from one-sixth of an hour to 1000 hours. The tests were made at the elevated temperatures. Loss of weight due to exposure to elevated temperatures was also determined.

It was observed that of the three types of laminate only the silicone bonded type retained enough strength to be tested after 10 hours' exposure to 700°F. However, the initial strength of the silicone bonded laminate at 73°F and at some of the lower elevated temperatures was much lower than that of the other two types of laminate. The phenolic bonded laminate gained weight after exposure to the lower range of elevated temperatures.

December 1953

SUBJECT: HIGH-TEMPERATURE CREEP-RUPTURE PROPERTIES OF GLASS-FABRIC-PLASTIC LAMINATES

INVESTIGATOR: John VanEcho, Gale R. Remely and Ward F. Simmons

CONTRACT: AF 33(038)-10818

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Creep and creep-rupture tests, in both tension and compression, were conducted at room temperature, 300°, and 500°F on three glass-fabric-base laminates. Resins used in these three materials were a silicone, DC-2104; a phenolic, CTL-91LD; and a polyester resin, PDL-7-669. Deterioration tests and some preliminary short-time tensile tests were also made on these three materials.

The creep and creep-rupture tests indicate that the PDL-7-669 material has the highest strength in tension at all three test temperatures of 80°, 300°, and 500°F. The highest compressive strength, on the other hand, was displayed by the CTL-91LD material under nearly all test conditions. However, both CTL-91LD and PDL-7-669 materials show a rapid decline in creep strength with time at 500°F. Beyond 50 hours' test time, the DC-2104 laminate displays the highest compressive creep-rupture strength at this temperature.

The short-time tensile tests show about the same strength relationship among the three test materials as do the creep-rupture tests. That is, the PDL-7-669 laminate is the strongest in tension and the CTL-91LD material appears to have superior strength in compression. These two materials, however, maintain their tensile strength superiority at 500°F possibly because they were held at the test temperature for only short periods of time.

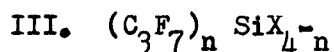
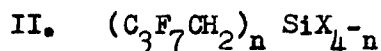
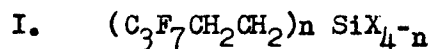
The deterioration-test results show that the DC-2104 silicone resin material is definitely the most stable of the three laminates after being exposed at 300° and 500°F for various periods of time. The long-time creep-rupture strength of these laminates at 500°F and perhaps 300°F, appears to be closely related to their high-temperature stability.

## RUBBER

TR 52-191, Part 2 (Uncl)

October 1953

SUBJECT: FLUORINE-CONTAINING ELASTOMERS  
INVESTIGATOR: O. R. Pierce and E. T. McBee  
CONTRACT: AF 33(038)-20581  
CONTRACTOR: Purdue Research Foundation  
ABSTRACT: The nature of the work described in this report comprises investigations of the synthesis of three types of fluorine-containing alkylsilanes:



X = Cl or OEt

Type (I) was prepared from the corresponding Grignard reagent and samples of two materials,  $(C_3F_7CH_2CH_2)Si(OEt)_3$  and  $(C_3F_7CH_2CH_2)_2Si(OEt)_2$  were submitted to the Materials Laboratory for polymerization studies.

The synthesis of type (II) was planned from the reaction of the corresponding fluorine-containing Grignard reagent or lithium reagent with silicon tetrachloride or tetraethyl silicate. The organometallic could not be prepared in good yield and the desired silanes were not obtained. Experimental details of the attempted synthesis are given.

The synthesis of type (III) was attempted from the reaction of either  $C_3F_7MgI$  or  $C_3F_7Li$  with silicon tetrachloride or tetraethyl silicate. The reaction products were found to be alkylsilanes but could not be identified conclusively.

TR 52-197, Part 3 (Uncl)

September 1953

SUBJECT: SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS  
INVESTIGATOR: F. A. Bovey  
CONTRACT: AF 33(038)-515  
CONTRACTOR: Minnesota Mining and Manufacturing Company  
ABSTRACT: This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants, and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Of the materials under development, the following appear to be of chief interest:

- A. Perfluorobutadiene Copolymers. Copolymers of perfluorobutadiene with 1,1-dihydroperfluoroalkyl vinyl ethers offer excellent high temperature resistance, low swelling in aircraft fluids, and high resistance to ozone.
- B. Fluoroacrylates. In addition to the 1,1-dihydroperfluoroalkyl acrylates, which have been described in earlier reports and which have outstanding solvent resistance but limited low temperature flexibility, newer classes of fluoroacrylates have been synthesized. Of particular interest are the  $\gamma$ -(perfluoroalkoxy)-1,1-dihydroperfluoropropyl acrylates, which offer solvent resistance at least equal to that of the earlier series but are flexible at temperatures 25°C. lower than the polymers of the 1,1-dihydroperfluoroalkyl acrylate.

In the compounding and vulcanization of fluoroacrylate polymers, the most striking accomplishment has been the development of polyamine curing systems which give greatly improved resistance to aircraft fluids such as diester lubricants at temperatures as high as 350°F., better compression set and other mechanical properties, and greatly improved resistance to nitric acid and to alkali.

Fundamental studies of the copolymerization behavior of the fluoroacrylates, of their swelling in organic liquids, and of their molecular weights are presented.

TR 53-223 (Uncl)

January 1954

SUBJECT: DEVELOPMENT OF LOW TEMPERATURE FUNGUS RESISTANT VINYL COMPOUNDS

INVESTIGATOR: Wallace W. Jackson

ABSTRACT: Efforts to develop a polyvinyl chloride plastic tubing conforming to and excelling the requirements of Specification MIL-I-7444 (Insulation, Sleeving, Electrical, Flexible) are described. This specification requires a combination of low temperature characteristics, fungus resistance and dielectric properties which commercial compounders have found difficult to obtain.

During this development a revised edition (MIL-I-7444-A) of this specification was made available. This revision requires flexibility at minus 90°F, but relaxes some of the other difficult requirements which were found to be unnecessary for most service applications.

Variation in compounding procedure and technique however, using a wide range of commercially available and experimental fungicides, plasticizers and stabilizers with a polyvinyl chloride/polyvinylidene chloride copolymer, resulted in a tubing compound exhibiting overall properties superior to those in the original version of Specification MIL-I-7444.

TR 53-281 (Uncl)

March 1954

SUBJECT: PLASTICIZERS FOR OIL-RESISTANT RUBBER  
CONTRACT: AF 33(616)-59  
CONTRACTOR: Phillips Petroleum Company  
ABSTRACT: A number of experimental plasticizers were prepared and evaluated as non-extractable plasticizers in oil-resistant rubber for service at low temperatures. These included n-formyl morpholine; diethylene glycol bis (morpholinylformate); diethylene glycol dicarbonate esters of methyl, ethyl, and butyl Carbitol; dibutyl and diisobutyl sebacate; bis (Methoxy Polyethylene Glycol 350) adipate; bis(2-methoxy-2-bromoethyl) phosphonate; formals prepared from ethyl Carbitol, Ethoxy Triglycol, the monoethyl ether of Polyethylene Glycol 200 and the mixed formal from Methoxy Polyethylene Glycol 350 and n-hexyl Carbitol; and the product from the reaction of butadiene monoxide with phosphorus pentoxide. A number of these experimental plasticizers imparted fairly good low temperature properties to Paracril B. Di-(Ethoxy Triglycol) formal was superior to Thiokol Corporation's TP-90B. However, none of these plasticizers combined the ability to impart low temperature properties with non-extractability to the degree desired. Of the plasticizers tested only n-formyl morpholine imparted fair low temperature properties combined with fair oil-resistance.

Thiokol Corporation's liquid polysulfide, ZL-109, was evaluated in combination with TP-90B. In compounding studies this material exhibited a strong tendency to scorch, but it was found that sulfur prevented the scorching and Altax retarded the cure. Promising polymers resulted from the addition of ZL-109 to a 70/30 butadiene-acrylonitrile latex.

A number of 1,3-butadiene-1-cyano-1, 3-butadiene copolymers were prepared and compared to Paracril B both with and without TP-90B and liquid polybutadiene as plasticizers. It was found that these experimental stocks were considerably inferior to Paracril B but had somewhat better low temperature properties. Temperature retraction and cold compression set data indicate a strong tendency for the cyanobutadiene stocks to crystallize.

TR 53-410 (Uncl)

January 1954

SUBJECT: INVESTIGATION AND DEVELOPMENT OF LOW-TEMPERATURE  
ADHESIVE TAPE

INVESTIGATOR: H. N. Homeyer, Jr. and J. H. Preston  
CONTRACT: AF 33(600)-16213  
CONTRACTOR: The Connecticut Hard Rubber Company  
ABSTRACT: This report describes work on the investigation and development of pressure-sensitive adhesives for application and use at sub-zero temperatures such as are encountered in certain aircraft and cold weather zone operations.

The research was carried out by personnel of the Connecticut Hard Rubber Company at New Haven, Connecticut during the period 9 April 1952 - 9 August 1953, under United States Air Force Research Contract AF 33(600)-16213.

The final product resulting from this work was twenty-five 60-yard rolls of two inch wide pressure-sensitive adhesive tape, meeting or exceeding all of the requirements of JAN-P-127 Type I, Grade A, and in addition being applicable and retaining adhesion down to -80°F. This tape has been forwarded to Wright Air Development Center.

The low temperature pressure-sensitive adhesive tape consists of silicone rubber adhesive compound, coated on a butyl rubber covered cotton print cloth backing.

TR 53-450 (Uncl)

May 1954

SUBJECT: DEVELOPMENT OF INTEGRAL FUEL TANK SEALANT COMPOUND  
INVESTIGATOR: John M. Snider and Frank Hirose  
CONTRACT: AF 33(600)-19202  
CONTRACTOR: Coast Pro-Seal and Manufacturing Company  
ABSTRACT: This work was undertaken to develop materials other than polysulfide polymers for use in fuel tank sealant applications. Materials for use in such applications preferably should be viscous liquid polymers which may be cured to an elastomeric state under ambient conditions after application.

Liquid polymers were prepared by the bulk copolymerization of acrylic esters and allyl glycidyl ether. These polymers could be cured to elastomeric products by the reaction of amine curing agents with epoxy groups. The cured products generally had good high temperature properties but poor low temperature flexibility. Resistance to aromatic fuels was not as good as would be desired.

Liquid polymers of a polyester type were prepared from mercaptoacetic acid. These polymers could be made

sensitive to oxidation curing reactions by a process of heat treatment. The cured polymers have excellent solvent and water resistance. The curing reaction is so rapid that it is difficult to mix the curing agent with the polymer and it will be necessary to develop suitable methods for retarding the reaction.

TR 54-68 (Uncl)

May 1954

SUBJECT: DEVELOPMENT OF AN IMPROVED IN-FLIGHT REFUELING HOSE  
INVESTIGATOR: Richard J. Meisinger, Stanley L. Bertholf and Frank Lichtey  
CONTRACT: AF 33(616)-386  
CONTRACTOR: United States Rubber Company  
ABSTRACT: A laboratory study was made to determine what type of plastic material would be suitable as a seamless tube for Aircraft In-flight Refueling Hose.

Of the various materials evaluated, FM-6901 type Nylon was chosen as the most suitable. A sample length of hose was manufactured using this material as an inner liner. The hose carcass used is identical to that now being furnished by the United States Rubber Company in 2-1/4" MIL-H-4495-A (ASG), Rubber, In-flight Refueling Hose which this company is qualified to manufacture. A nylon tube was extruded and incorporated into the carcass using special techniques developed for this purpose.

The resultant hose sample was subjected to the low temperature test required by the contract and failed to meet requirements. The nylon liner cracked during bending.

It is concluded from this work that even though Nylon FM-6901 in strip specimens is flexible at -65°F it is unsatisfactory at this temperature when used in a hose.

#### SANDWICH CONSTRUCTION

TR 52-51, Part 2 (Uncl)

September 1953

SUBJECT: FOAMED METAL LOW DENSITY CORE MATERIAL FOR SANDWICH CONSTRUCTION  
INVESTIGATOR: Johan Bjorksten, John C. Elliott and Robert J. Roth  
CONTRACT: AF 33(038)-21838 S1 (52-126)  
CONTRACTOR: Bjorksten Research Laboratories, Inc.  
ABSTRACT: Progress on the development of an extrusion process for the preparation of metallic low density foams from magnesium-aluminum alloys is described. Difficulties were encountered in a

number of process variables and further work is required to develop a successful process.

Inasmuch as this work is being continued, any conclusions must be regarded as tentative and subject to modification based on additional investigation.

TR 52-51, Part 3 (Uncl)

May 1954

SUBJECT: FOAMED METAL LOW DENSITY CORE MATERIAL FOR SANDWICH CONSTRUCTION  
INVESTIGATOR: Johan Bjorksten, John C. Elliott and Robert J. Roth  
CONTRACT: AF 33(038)-21838 S3 (53-591)  
CONTRACTOR: Bjorksten Research Laboratories, Inc.  
ABSTRACT: Research and development work proving the feasibility of foaming metals by means of a miscible gas former and progress in the design and construction of prototype equipment for the continuous production of low density foams from aluminum-magnesium alloys are described. The most important innovations in the above phases of work performed subsequent to the work described in WADC Technical Report 52-51 Part 2 are improved quality of foam through the introduction of air or oxygen into the foamed mixture prior to its solidification and the introduction of aluminum oxide-coated steel, a material with high corrosion resistance towards molten aluminum, magnesium, and their alloys, as a new structural material for certain components of the foaming equipment. Further improvement in quality and uniformity of product is seen to be largely contingent upon the construction of pilot plant equipment capable of providing a more truly continuous flow of foam in larger quantities.

TR 52-184, Supplement 1 (Uncl)

October 1953

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT  
INVESTIGATOR: Donald G. Coleman  
CONTRACT: AF 33(038)-51-4326E  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Developments in the program of research in sandwich aircraft construction conducted by the U. S. Forest Products Laboratory during fiscal year 1953 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Five technical reports issued during the fiscal year are abstracted.

TR 53-146 (Uncl)

December 1953

SUBJECT: DEVELOPMENT OF A HEAT-RESISTANT FOAMED-IN-PLACE LOW-DENSITY SILICONE RESIN CORE MATERIAL  
INVESTIGATOR: Kenneth R. Hoffman and Donald E. Weyer

CONTRACT: AF 33(600)-6320  
CONTRACTOR: Dow Corning Corporation  
ABSTRACT: Methods for producing low density silicone resin core material have been developed. The foams are unicellular and have excellent heat stability along with low moisture absorption and good electrical properties.

The resins, at 100% solids, were expanded by the thermal dissociation of a blowing agent blended with the resin. None of the materials used in foaming was toxic.

Numerous silicone resins were evaluated and the revised specifications of the contract were met by using Resin XR-544. The expanded resin gave a large variety of physical properties. Controlled density ranged from 8 to 24 pounds per cubic foot and compressive strength varied from 90 to 850 psi at room temperature. Compressive strength at 500°F after 1/2 hour exposure, was from 20 to 240 psi. Thermal life of the foams was over 300 hours at 500° and 600°F with no appreciable weight loss or change of appearance. Specifically, a foam with a 17 lb./cu.ft. density had a room temperature compressive strength of 425 psi. After 1/2 and 200 hours at 500°F its compressive strength was 125 psi and 115 psi respectively. Several of the foams were exposed to 700°F for 10 hours without qualitative change. They were non-flammable and very resistant to a direct flame.

The resins were also foamed -in-place between glass fabric base silicone resin laminated skins. Although comparatively little work has been completed on this phase of foaming, it appeared that the core was fairly uniform and bonded to the faces.

TR 53-327 (Uncl)

December 1953

SUBJECT: DEVELOPMENT OF PLASTIC EXPANDED PELLET-TYPE CORE MATERIAL FOR SANDWICH CONSTRUCTION  
INVESTIGATOR: Arthur Derr Golladay  
CONTRACT: AF 33(600)-19090  
CONTRACTOR: Golladay Aeronautical Laboratory  
ABSTRACT: Preliminary work has been conducted on expanded plastic pellet-type, low density core materials for fabricating sandwich construction. Expanded plastic pellets of a wide range of sizes have been developed and produced on a laboratory basis by methods closely identical with mass production techniques.

This program has been concerned with the development of core materials making use of commercially available resins, not with the development of basic materials. However, some resin investigation has been necessary, but only enough necessary to produce pellets and core compositions suitable for demonstration of the pellet type core material.

Pellet-type core material weighing 10 pounds per cubic foot has been produced which resulted in a maximum compressive strength of 166 psi. at 2% strain. Other formulations investigated varied from 18 to 96 psi. In comparison to other presently available plastic core materials the present pellet type has low compressive strength. Based on 10 pounds per cubic foot density, the pellet core has less than 46 and 10 percent of the ultimate compressive strength of commercially available foamed-in-place and honeycomb cores, respectively.

#### TEXTILES

TR 53-21, Part 2 (Uncl)

July 1953

SUBJECT: A STUDY OF THE EFFECT OF TEMPERATURE ON TEXTILE MATERIALS  
INVESTIGATOR: Myron J. Coplan and Eric Singer  
CONTRACT: AF 33(038)-22932  
CONTRACTOR: Fabric Research Laboratories, Inc.  
ABSTRACT: A group of fabricated textile materials including seam samples were tested at four specific temperature conditions. The materials involved included cotton in addition to the nylon materials. The samples included thread and suspension line as well as cloth. The seam samples were constructed as called out in parachute fabrication drawings for two needle seams, and skirt butterfly attachments.

The strength of several nylon materials was found to vary with temperature. It was found that the sensitivity to temperature is due in part to geometric influences on the inherent yarn strength. During the preheating of the nylon materials, sufficient dehydration took place causing a loss in efficiency at 210°F.

As expected, the cotton materials lost considerable strength at the low and high temperatures.

A number of specific conditions must exist for sticking of nylon cloth to nylon cloth to be evidenced. Indications are that prolonged exposure to heat (350° to 370°F) may reduce the cohesion of the two surfaces. Therefore sticking will not be caused due to temperature over a certain period of time. Conditions such as sharp impact, surface over surface shear at high pressures may result in some sticking or cohesion at lower temperatures.

December 1953

SUBJECT: INVESTIGATION OF MYLAR-TYPE POLYESTER FILMS FOR PARACHUTE CANOPIES  
INVESTIGATOR: Johann Bjorksten, Risto P. Lappala and Robert J. Roth  
CONTRACT: AF 33(616)-403  
CONTRACTOR: Bjorksten Research Laboratories, Inc.  
ABSTRACT: Double layer Mylar films reinforced with Dacron fiber have exhibited promising properties for use in parachute canopies. These properties include:

Tensile Strength	82 - 90 pounds
Burst Strength	195 - 220 pounds
Weight Per Square Yard	2.44 - 4.42 ounces
Thickness (maximum)	0.007 - 0.008 inches
No embrittlement at -60°C. and retention of useful properties at 150°C.	

These films were prepared by cementing a reinforcing grid of 2, 3 or 4 ply Dacron thread between two 50 or 100 gauge Mylar films.

Miniature parachutes made of Reinforced Mylar Films had exceptional resistance to tearing, to failure by flexing or creasing, and to abrasion. Accelerated rapid-load tests of these parachutes did not produce any failure in the seams or material. Reinforced Mylar Film thus compared favorably with Nylon except for its weight and porosity which could probably be improved by further investigation.

TR 53-351 (Uncl)

September 1953

SUBJECT: DEVELOPMENT OF 0.9-OZ. NYLON PARACHUTE CLOTH  
INVESTIGATOR: H. J. Bickford  
CONTRACT: AF 33(600)-23196  
CONTRACTOR: Cheney Brothers  
ABSTRACT: A 4,000-yard lot of Nylon Parachute Cloth weighing 0.9 ounce per square yard or less was woven and finished with Silicone Oil. The properties of this fabric were tested and found to meet the target properties laid out in the contract.

Accelerated aging tests indicated that the Silicone Oil provides a measure of protection against fabric deterioration and definitely improves the tear resistance at all times.

This fabric can fulfill certain experimental purposes where an extremely light thin parachute fabric is needed for models. In addition the material could be used in critical applications

requiring small yardage. However, the difficulties of manufacture are so extensive that use of this fabric should be confined to special applications which justify its use.

TR 53-444 (Uncl)

November 1953

SUBJECT: EVALUATION OF EXPERIMENTAL WOOL AND SYNTHETIC  
BLENDS IN AIR FORCE VELOUR OVERCOAT MATERIAL  
INVESTIGATOR: Ormond J. Roberts, 1st Lt, USAF and C. A. Willis  
ABSTRACT: A group of 30 wool/synthetic, blended velour over-  
coat fabrics were evaluated to determine the effects on fabric proper-  
ties when the fiber and composition was varied.

The synthetic fibers involved included Orlon, Dacron, Acrilan, Viscose and Dynel. Each synthetic fiber was blended with wool in percentages of 10, 20, 30, 40, 50 and 60 to make a series of 6 samples. One sample was fabricated of 100 percent wool to be used as a control.

The use of Orlon in the fabric increased the warp and filling breaking strength and flat and flex abrasion resistance. The shrinkage in sponging and crease resistance were not significantly affected.

The use of Dacron in the fabric increased the breaking strength. Flex abrasion resistance was increased when the synthetic was used in percentages of 20 or above. Flat abrasion resistance was not significantly affected. Shrinkage in sponging and crease resistance was not appreciably affected.

The use of Acrilan in the fabric increased the breaking strength when used in percentages of 50 and above. Reduced breaking strength was noted when 10 and 20 percent Acrilan was used. Resistance to abrasion, both flat and flex, was considerably lower in all percentages, and remained approximately equal throughout the entire series of samples. Crease resistance and shrinkage in sponging were not significantly affected.

The use of viscose in the fabric decreased the breaking strength and flex and flat abrasion resistance. Crease resistance was approximately equal to the all wool control. Shrinkage in sponging was slightly higher in the warp direction with a gain noted in the filling direction.

The use of Dynel in the fabric increased the breaking strength when used in percentages above 10. Resistance to abrasion, both flat and flex, was markedly decreased. Shrinkage in sponging and crease resistance were not significantly affected.

As the percentage of synthetic was increased in the blend the difficulties encountered at the fulling process increased and it became more difficult to obtain the desired color and finish.

Due to the difficulties encountered in processing velour fabrics using wool and synthetic fibers, the Air Force will continue to use only the 100% wool fabric. In the event of an emergency and as a wool conservation measure, synthetic fibers are recommended only in the amounts up to 20 percent.

TR 53-452 (Uncl)

May 1954

SUBJECT: DEVELOPMENT OF DYE FORMULATIONS AND DYEING PROCEDURES FOR PRODUCING SAGE GREEN PILE FABRIC

INVESTIGATOR: A. Bartovics

CONTRACT: AF 33(600)-23440

CONTRACTOR: The George W. Borg Corporation

ABSTRACT: Dye formulations and dyeing procedures are presented for producing the required shade of Sage Green Number 530 with the maximum obtainable colorfastness properties for Type I (Acrilan) and Type II (Vicara-Dynel) pile fabric conforming to Specification MIL-C-6590. Physical and colorfastness properties of the respective fabrics thus produced are tabulated, and are shown to conform to the requirements of the above specification.

A tentative dye formulation and dyeing procedure for producing Sage Green Type III (Orlon) fabric and the physical properties of a preliminary sample of this fabric are also reported. This covers the work done on Type III fabric up to the date of its suspension from the contract by the USAF.

TR 54-12 (Uncl)

March 1954

SUBJECT: THE EFFECT OF A SYNTHETIC LUBRICANT AND TWO DI-BASIC ACID ESTERS ON CERTAIN USAF FABRICS

INVESTIGATOR: Clarence D. Smith, 1st Lt, USAF

ABSTRACT: This investigation was initiated to determine if fabrics employed by the USAF were subject to degradation or deterioration when exposed to the lubricating oil covered by Specification MIL-L-7808 and the acid esters, di-2-ethyl hexyl adipate and di-2-ethyl hexyl sebacate. Need for an investigation was realized upon receipt of information that fabrics had been damaged when exposed to synthetic lubricants. In order to accomplish the desired program a series of fabrics composed of fibers that are commonly used in USAF fabrics was exposed to the above synthetic lubricants at room temperature and at 160°F. After exposure, laundering and dry

cleaning tests were conducted to determine their effect in conjunction with the lubricants. By diaphragm burst tests it was found that no damage was apparent in the fabrics used when exposed to the above synthetic lubricants.

#### TRANSPARENT MATERIALS

TR 6684 (Uncl)

December 1953

SUBJECT: FORMING TEST FOR TRANSPARENT PLASTIC SHEET MATERIALS  
INVESTIGATOR: E. N. Robertson and C. H. Weber  
CONTRACT: AF 33(038)-22945  
CONTRACTOR: Rohm and Haas Company  
ABSTRACT: Development of a test procedure and apparatus for use in qualification testing of transparent plastic sheet material under military specifications is described. The procedure was used to differentiate the forming characteristics of five transparent plastic sheet materials that are supplied under five different Government Specifications for use in aircraft enclosures.

The plastic materials were found to be sufficiently different in characteristics at high temperatures to have distinctly different moduli of elasticity and to require different conditions of temperatures and pressures for forming. The forming parameters (thickness, pressure, time and temperature) which will permit qualification testing to differentiate the materials are given.

TR 52-185, Supplement 1 (Uncl)

December 1953

SUBJECT: DEFLECTION AND DISTRIBUTION OF STRESSES IN THE FACINGS OF A CENTRALLY LOADED TRANSPARENT SANDWICH BEAM  
INVESTIGATOR: Wilhelm S. Ericksen  
CONTRACT: AF 18(600)-70 CONTRACTOR: Forest Products Lab.  
ABSTRACT: Formulas for the deflection and for the stress within the facings of a transparent sandwich beam under central load were presented in the basic issue of this report. However, photos and figures pertinent to the calculations were omitted from that report. These photos and figures are included herein as a supplement to the original issue.

TR 53-19 (Uncl)

September 1953

SUBJECT: DEVELOPMENT OF A PRACTICAL LABORATORY PROCEDURE TO BE USED IN EVALUATING THE FORMING QUALITIES OF PLASTIC SHEET MATERIALS  
INVESTIGATOR: Paul H. Kaar

CONTRACT: AF 33(038)-27648  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: Under the terms of the contract with Wright Air Development Center, the purpose of this research project was (1) to study the various factors which are of importance in the fabrication of flat plastic sheet material into useful shapes for a transparent aircraft enclosure, and (2) to develop a practical laboratory procedure for evaluating the forming qualities of these plastic materials. In order to achieve this objective, it was necessary to review the industrial processes and applications of the material and to determine what measurable characteristics are important in forming operations.

Generally, the investigation of a formability criterion proceeded along two separate lines. One approach was an attempt to use various standardized engineering test procedures to indicate formability. The other approach was to duplicate various manufacturers' forming operations and use data derived from these tests to evaluate forming characteristics. Apparatus development was a significant part of each approach.

This final report includes discussions and data pertinent to the selection of the formability criterion recommended and a discussion of the important factors in formability evaluation.

The formability rating system recommended consists of forming, by positive air pressure, an unconfined bubble of the plastic heated to optimum forming temperature. The rating assigned is based on the pressure required to form the bubble and the extent to which it can be formed before fracture. Plastics incapable of being stretched are rated by a bend test. Conclusions reached are as follows:

1. A suitable formability criterion for transparent plastic sheet must embody evaluation of (1) ease of forming and (2) maximum extent of forming possible.
2. Standardized engineering tests performed at optimum forming temperature and providing such data as maximum elongation, creep, and ball penetration values fail to evaluate formability satisfactorily for the following reasons:
  - a. Data from such tests do not correlate satisfactorily with ad hoc tests duplicating manufacturing operations.
  - b. Engineering tests do not provide sufficient spread in results to distinguish between plastics of similar forming properties.
3. These tests have shown that several systems can be used to evaluate the two primary formability characteristics

of plastic sheet. Any of the ad hoc tests, particularly the hemispherical draw and unconfined bubble tests, can be used to evaluate the modulus of workability. The deformative maximum is more difficult to gage; only the unconfined bubble test using a lubricated specimen was judged to measure impartially this property of the plastic.

TR 53-259 (Uncl)

September 1953

SUBJECT: F-94 ONE HALF SCALE METHYLALPHACHLORACRYLATE CANOPY  
INVESTIGATOR: Frank Evans, M. Elber Latham and John G. Stansbury  
CONTRACT: AF 33(616)-202  
CONTRACTOR: Swedlow Plastics Company  
ABSTRACT: Quarter-inch thick Methylalphachloracrylate monolithic cast sheet was satisfactorily fabricated by a "free blowing" technique into a half scale F-94 canopy. The optical quality was excellent and in every way appeared to be easily the equivalent to that of a "free blown" acrylic part (MIL-P-6886 or MIL-P-5425). However, some surface degradation was evidenced by a slight darkening. An adequate cement, as well as cementing technique, was established to assemble the edge attaching materials to the formed Methylalphachloracrylate canopy.

TR 53-299 (Uncl)

December 1953

SUBJECT: PRELIMINARY EXPLORATION OF SILICON-FLUORIDE PLASTICS FOR HIGH-TEMPERATURE-RESISTANT INTERLAYERS  
INVESTIGATOR: Johan Bjorksten, Luther L. Yaeger, Robert P. Cox and Robert J. Roth  
CONTRACT: AF 33(600)-23256  
CONTRACTOR: Bjorksten Research Laboratories, Inc.  
ABSTRACT: The objective of this study was a preliminary exploration of the possibilities of silicon-fluoride plastics for high-temperature-resistant interlayers. It was understood that the investigation was not to give a final and conclusive result, but rather to cover a broad scope to develop promising leads and approaches.

The following were explored experimentally and yielded transparent products with good temperature stability and high resistance to di-ester type lubricants and aromatic solvents:

1. Cross-linking a fluoroalkyl acrylate with a polysiloxane.
2. Co-polymerizing a fluorinated olefin and an unsaturated silane.

A third approach, preparing a fluoro-silicon analogue of a polyester resin, yielded an intermediate of promising appearance.

Samples of these materials have been submitted to WADC for evaluation.

II. TECHNICAL REPORTS 1 JUNE 1951 - 30 JUNE 1953 (NOT ABSTRACTED)  
(Not listed in WADC TR 53-373)

ADHESIVES, STRUCTURAL

TR 5896, Part 2 (Uncl)

July 1951

SUBJECT: HIGH-TEMPERATURE METAL-TO-METAL ADHESIVES  
INVESTIGATOR: A. E. Smith, W. C. Imholz and P. M. Elliott  
CONTRACT: W33-(038)-ac-20810(20050)  
CONTRACTOR: U. S. Rubber Company

ALLOYS, HIGH TEMPERATURE

TR 52-54 (Uncl)

March 1952

SUBJECT: MALLEABLE CHROMIUM AND ITS ALLOYS  
INVESTIGATOR: Earl T. Hayes  
CONTRACT: AF 33(038)-50-1084-E  
CONTRACTOR: U. S. Bureau of Mines

ALLOYS, NONFERROUS, TITANIUM

TR 6597, Part 2 (Uncl)

March 1952

SUBJECT: THE TITANIUM-IRON PHASE DIAGRAM  
INVESTIGATOR: W.J. Fretagne, C.S. Barker and E.A. Peretti  
CONTRACT: AF 33(038)-8495  
CONTRACTOR: University of Notre Dame

TR 6623 (Uncl)

June 1951

SUBJECT: DEVELOPMENT OF TITANIUM-BASE ALLOYS  
INVESTIGATOR: J. W. P. Rengstorff, C. T. Greenidge, L. W. Eastwood,  
C. H. Lorig, J. O. Brittain, H. A. Pray, P. D. Frost,  
W. H. Kearns, R. D. Williams, C. B. Voldrich,  
W. F. Fink, R. F. Peoples, W. F. Simmons, and H. C. Cross  
CONTRACT: AF 33(038)-3736  
CONTRACTOR: Battelle Memorial Institute

ALLOYS, NONFERROUS, ZIRCONIUM

TR 52-236 (Uncl)

November 1952

SUBJECT: U. S. BUREAU OF MINES ZIRCONIUM ALLOY INVESTIGATION  
INVESTIGATOR: Earl T. Hayes, R. L. Carpenter, A. D. Cavett, H. Kato,  
W. L. O'Brien and O. G. Paasche  
CONTRACT: AF 33(038)-50-1214-E  
CONTRACTOR: U. S. Bureau of Mines

ANALYSIS AND MEASUREMENT

TR 6381 (Uncl)

November 1952

SUBJECT: AN EXAMINATION OF SOME AIRCRAFT GREASES WITH THE  
ELECTRON MICROSCOPE  
INVESTIGATOR: Jules I. Wittebort and Bernard Rubin  
CONTRACT: RDO 601-299

TR 6610 (Uncl)

December 1951

SUBJECT: COMPOSITIONAL ANALYSIS OF AN-G-25 TYPE GREASES  
INVESTIGATOR: Nora E. Funkhouser and George Rappaport  
CONTRACT: E. O. No. R601-299

BIOCHEMISTRY

TR 6518, Part 3 (Uncl)

November 1952

SUBJECT: FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COM-  
POUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 3 -  
FLUORINATED ANISOLES, BENZYLs, BENZOIC ACIDS,  
BIPHENYLS, PHENOLS, AND TOLUENES  
INVESTIGATOR: Leo Roy Tehon  
CONTRACT: AF 33(038)-10897  
CONTRACTOR: Illinois State Natural History Survey

CRITERIA, DESIGN

TR 52-89, Part 3 (Uncl)

June 1953

SUBJECT: PLASTIC BEHAVIOR OF ENGINEERING MATERIALS. PART 3 -  
CRITICAL REVIEW AND INTERPRETATION OF THE LITERATURE  
ON PLASTIC (INELASTIC) BEHAVIOR OF ENGINEERING METALLIC  
MATERIALS.

CRITERIA, DESIGN CONT'D

INVESTIGATOR: M. C. Steele, C. K. Liu and J. O. Smith  
CONTRACT: AF 33(038)-15677  
CONTRACTOR: University of Illinois

ELECTRODEPOSITION

TR 52-232 (Uncl)

December 1952

SUBJECT: ELECTRODEPOSITION OF TITANIUM FROM AQUEOUS SYSTEMS  
INVESTIGATOR: Ernest J. Breton, 1st Lt, USAF  
CONTRACT: RDO No. 611-11

PETROLEUM PRODUCTS, ANTISEIZE COMPOUNDS

TR 52-102 (Uncl)

September 1952

SUBJECT: DEVELOPMENT OF TEST METHODS FOR ANTI-SEIZE COMPOUNDS  
INVESTIGATOR: J. W. Cunningham  
CONTRACT: AF 33(038)-22805  
CONTRACTOR: Southwest Research Institute, San Antonio, Texas

PETROLEUM PRODUCTS, FUELS

TR 52-53 (Uncl)

September 1952

SUBJECT: STUDIES TO DETERMINE THE ELECTRICAL AND PHYSICAL  
PROPERTIES OF AVIATION FUEL  
INVESTIGATOR: C. C. Petersen  
CONTRACT: AF 33(038) 3793  
CONTRACTOR: Armour Research Foundation

PETROLEUM PRODUCTS, HYDRAULIC FLUIDS

TR 53-79 (Uncl)

May 1953

SUBJECT: CHLORINE AND FLUORINE CONTAINING COMPOUNDS FOR  
NONFLAMMABLE MATERIALS  
INVESTIGATOR: Ogden R. Pierce and Earl T. McBee  
CONTRACT: AF W33-038 ac-19024  
CONTRACTOR: Purdue University

PETROLEUM PRODUCTS, LUBRICANTS

TR 52-220 (Uncl)

September 1952

SUBJECT: STUDIES TO DETERMINE THE ELECTRICAL AND PHYSICAL  
PROPERTIES OF AIRCRAFT LUBRICATING OILS  
INVESTIGATOR: Joseph L. Radnik  
CONTRACT: PO 33(038)-3793  
CONTRACTOR: Armour Research Foundation

TR 53-11 (Uncl)

April 1953

SUBJECT: LOW-TEMPERATURE LUBRICATING ENGINE OIL ADDITIVES  
INVESTIGATOR: Gilbert Gavlin, Edwin A. Swire and Sam P. Jones, Jr.  
CONTRACT: AF 33(038)-1644  
CONTRACTOR: Armour Research Foundation, Illinois Institute of  
Technology

RUBBER

TR 52-80

November 1952

SUBJECT: INVESTIGATION OF PLASTICIZERS FOR OIL-RESISTANT  
RUBBER FOR SERVICE AT LOW TEMPERATURES IN CONTACT  
WITH HYDROCARBON FLUIDS  
INVESTIGATOR: J. C. Hillyer and C. S. Imig  
CONTRACT: AF 33(038)-17201  
CONTRACTOR: Phillips Petroleum Company

TRANSPARENT, MATERIALS

TR 52-185

August 1952

SUBJECT: DEFLECTION AND DISTRIBUTION OF STRESSES IN THE FACINGS  
OF A CENTRALLY LOADED TRANSPARENT SANDWICH BEAM  
INVESTIGATOR: Wilhelm S. Ericksen  
CONTRACT: AF 18(600)-70  
CONTRACTOR: Forest Products Laboratory, Forest Service, U. S.  
Department of Agriculture

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